# Development of a Polymetric Grout for the Hydrostatic Bearing at DSS 14

C. E. McClung, J. L. Schwendeman, G. L. Ball III, and G. H. Jenkins
Monsanto Research Corporation

R. D. Casperson, G. P. Gale, and A. A. Riewe
DSN Engineering Section

*The grout under the hydrostatic bearing runner at DSS 14 has exhibited a rapid rate of deterioration and premature failure. This report examines some of the probable causes of the deterioration. It also describes the development of a polymetric grout that is more resistive to the causes of early grout failure.*

## I. Introduction

### A. Geometry

The hydrostatic thrust bearing of the 64-m antenna consists of a steel runner 12.7 cm thick, 24½ m outside diameter and 22½ m inside diameter, and three pads each 16258 $cm^2$ in area, each carrying $2.75 \times 10^6$ kg (see Figs. 1 and 2). High-pressure oil is forced between the lower surface of the pads and the top surface of the runner enabling the antenna to rotate on a thin film of oil approximately 0.25 mm thick. Because the film thickness between the bottom of the pads and the top surface of the runner is so thin, the top surface of the runner must be maintained in a very flat condition. Any degradation in the grout that supports the runner will cause the runner surface to warp into a nonflat condition with a resulting decrease in the oil-film thickness separating the bearing surfaces.

### B. History

Initially the runner was precisely leveled and Embeco grout was poured between the bottom of the runner soleplate and top of the concrete pedestal. The Embeco grout contained a corrosive additive that rusted the underside of the soleplate. Soon both the Embeco and soleplate had to be replaced, this time with a thicker soleplate and a drypack grout consisting of portland cement and sand with only enough moisture to provide for bonding during placement.

However, the deterioration of the flatness of the hydrostatic bearing runner flatness, although significantly slower, persisted. It appeared to be worse in areas where bearing oil leaked from the runner joints and reservoir seals. Particles of cement and sand could be observed being flushed out of the grout with the oil leakage. Samples of oil soaked grout were subjected to compression tests and it was found that once the portland cement grout had thoroughly cured, oil did not appear to reduce the strength enough to cause the degradation we were observing.

While searching for plausible causes for the grout deterioration and for possibly a different grout material that would stand up to our rigid requirements, we contacted several

knowledgeable organizations, and finally authorized a contract to Monsanto Research Corporation, Dayton Laboratory.

This article contains much information and findings supplied by George L. Ball III, George H. Jenkins, Charles E. McClung, and James L. Schwendeman of Monsanto Research, Inc., as reported in their periodic and final reports. The work was planned for completion in two phases. Phase 1 was directed toward screening and testing five generic types of materials believed to be potentially able to meet these needs. The physical properties, strength, modulus of elasticity, and resistance to erosion and chemical attack by the oil were to be evaluated, and any unique requirements imposed by each material for mixing, placing, compacting, and cooling (where high heat of polymerization might be a factor) were to be identified. The planned objective of Phase 1 was to identify the single generic type of material best suited to replace the dry pack grout. Phase 2 consisted of developing, refining, and optimizing the selected material to achieve a high-strength, durable grout replacement.

Work in Phase 1 began in July 1978 with a visit to the Goldstone antenna site. The complete repair process—removal of the deteriorated grout, cleaning and preparation of the exposed surfaces, mixing and repacking with the dry pack, and leveling the repaired area—provided a familiarity with the service environment and the repair facilities.

## II. Technical Discussion

### A. Analysis of the Problem

To design a more durable antenna grout, the events and stresses leading to the early failure of the dry-pack material must be understood. Some of these factors were not identified until late in this work. However, based upon data on the track displacement during rotation, the measured physical properties of the grout, and work with the dry-pack material used to repair deteriorated sections, the early failure problem now appears to be caused by four separate, but closely interrelated factors:

(1) The flexing motion of the track as the antenna rotates is probably the initiating factor in the grout failure. With rotation, the track rises in front of each leg, collecting a pond of oil between the track and grout. As the leg passes, the track and, to some extent, the sole plates, move down, producing a high-pressure oil flushing action that penetrates and erodes the grout surface. Samples taken from the track interface at the Goldstone site show definite evidence of this erosive action.

(2) The hydration reaction of cure of a portland cement is dependent on the sustained contact of the reacting cement particles with water. When the leaking oil reaches and wets the concrete surface, further bonding and strength development is stopped. This probably happens in some areas during or very shortly after the placement of a fresh grout repair. Thereafter, the weakly bonded or nonbonded surface aggregates would be most vulnerable to erosion by the flushing oil.

(3) The materials and mix design now used produce a strong, dense, high-strength grout when the design moisture content is maintained, and when oil contact is prevented during cure. But the mix has little more than the stoichiometric amount of water needed for cure, and the grout is very vulnerable to damage by evaporative water loss during mixing and placing. Evidence of this was seen during the preparation of the control specimens when, after four days cure, the test cylinders were easily broken at the junction of each packed increment of grout. The cure-arresting effect of oil leaking into the grout would be synergistic, selectively penetrating and wetting the dry, water-poor zones. This combination of events would severly damage the grout/sole-plate surface and particularly the material at the packing increment divisions, and hasten erosion by the pumping oil.

(4) Under ideal conditions, the slow rate of cure by types I or 2 portland cement may not allow the grout to reach load-bearing strength within the time allotted for repairs.

### B. Portland Cement Dry-Pack Grout

The preparation of dry-pack grout test specimens was included in this program to provide a basis for comparison and evaluation of the candidate replacement materials. The vulnerability of portland cement to surface coating by the leaking oil before cure was known, but its sensitivity to evaporative water loss and the extent to which this loss inhibited normal curing and accelerated damage by the oil was first noted during the preparation of the dry-pack test cylinders.

**1. Water loss.** Water lost from the dry-pack grout mix at two steps in the repair process may increase its vulnerability and accelerate damage done by the leaking oil. The dry-pack mix design contains adequate water for hydration. However, the 8.4% water in the mix is largely in the form of a surface coating on the particles. Because of the high surface area, the mix is very subject to evaporative drying. Some water is necessarily lost in the process of rescreening after mix to eliminate lumps. Then, with a reduced water content, the mix

is highly vulnerable to further exposure and water loss such as occurs at the surface of each increment placed and compacted. If the water content at these exposed surfaces becomes less than that needed for hydration of cement and if the overall water content has been sufficiently reduced to preclude migration from interior wetter zones, the interfaces cannot cure properly. Note: Our water content is determined by behavior of grout "as placed" not by "as mixed" performance. Thereby significantly reducing the effects of water loss.

In the laboratory environment (RH = 50%) water loss during the delumping process was found to be as high as 5% to 6% of the total water. At the site, with longer exposure times and lower humidity, the loss could be much greater. The effects of the water loss are shown in Fig. 3. Each increment packed in a 7.62-cm-diameter cylinder mold was clearly delineated, and very little bonding developed between the sections. On impact, the cylinder broke cleanly at the division, and imprints of the packing ram were clearly visible on the fracture surface. By contrast, an impact fracture within a single packing increment in this cylinder required a much greater impact force, and the fracture path was random in direction, typical of a sound concrete.

The dry, nonbonded interfaces seen in this cylinder would be readily wetted by the leaking bearing oil and thus would be subject to erosion. This condition could, in fact, be the origin of the channels (Fig. 4) observed at the grout/sole-plate interface in the deteriorated grout removed from the Goldstone site.

To determine the effect of water loss on curing rate, two batches of the dry-mix grout were prepared under identical conditions except that, with the first batch, the evaporative water loss was measured and corrected to a water-to-cement ratio (w/c) of 0.23 just before placement. The water lost from the second batch (w/c = 0.213) was not replaced. Two 16.4-$cm^3$ samples prepared from these mixes were tested for development of compressive strength with time over a 48-hour cure period. Three specimens were tested at each interval. The data are shown in Fig. 5.

The average compressive strengths of the two mixes were similar through the 8-hour test interval. Between 8 and 16 hours, however, the rate of strength development by the low-water mix fell off sharply. The strength of these specimens averaged about 70% of the water-corrected mix, and the disparity increased with time through the 48-hour test.

The variations from specimen to specimen seen here after identical curing times may be more significant than the average differences in strength of the two mixes. With the higher water (corrected) mix, the individual test values at all intervals were within $1.03 \times 10^6$ $N/m^2$ of the average, and the maximum variation occurred, as would be expected, during the periods of most rapid strength gain, between 8 and 24 hours. At other test intervals, the individual specimen values were within $6.9 \times 10^5$ $N/m^2$ of the average.

The data scatter observed was much greater with the low-water specimens, and reached almost $6.9 \times 10^6$ $N/m^2$ at 48 hours. Most significantly, this variation from sample to sample increased with cure time. The increasing scatter with time suggests a nonhomogeneous composition, some parts of which are curing normally while others (dry areas) are developing strength at a much lower rate or not at all.

The water-corrected mix gained strength rapidly, and probably became load-bearing $5.9 \times 10^6$ $N/m^2$ in about six hours. By contrast, the non-water-corrected specimens typically required about 8 hours, but some of these specimens at 8 hours were well below load-bearing strength. Thus movement of the antenna at such a time would have caused overloading and compressive failure in the grout.

**2. Oil erosion tests.** A test fixture was designed and built to simulate the erosive action of high-pressure oil flow believed to be a major contributor to the grout failure. The fixture was used to cyclically create a high-pressure oil flow across a 7.62-cm-diameter surface of the test cylinders and, at the end of each cycle, to apply a $2.07 \times 10^7$ $N/m^2$ compressive load. When charged with the bearing oil, the piston load is hydraulically transmitted to the sample surface. Limited to one exit port 16 mm in diameter, the compression stroke of each cycle caused a high-velocity, unidirectional oil flow over the sample surface. Oil flowing across the concrete approached a velocity of about 1000 km/h as it entered the port.

The portland cement dry-pack grout, shown in Fig. 6a after 15,500 cycles, shows only minor damage by erosion. Some minor pitting and erosion was seen near the outside rim of the specimen and mostly around the exit port area, which is the area of greatest volume movement. The pitting is shallow, and in most cases, single, small aggregate particles were lost from the outer surface. No deep channels or structural damage was seen. It is not believed that erosion to this limited extent would adversely affect the antenna operation.

Most importantly, this sample was monitored for evaporative water loss during mixing, and the water content was corrected (to w/c = 0.23) just before placement. Figures 6b and 6c show the results of placing and packing the test surface in two increments, such that a packing interface was created parallel to the direction of oil flow. Here the water lost during mixing was not replaced, and the test surface was exposed to oil after 8 hours cure. The total cure time before start of the

erosion tests was seven days, similar to other erosion test specimens.

Figure 6b shows the test surface after 5,700 cycles. Severe erosion all along the packing interface was seen and in some areas had reached depths of 5.1 mm. (Note: the halo effect on this picture is the out-of-focus oil retention sleeve, left in place for continued testing.)

Figure 6c shows the sample after 13,700 cycles. Erosion had then become so severe that testing was discontinued. The channel width had grown to 12.7 mm in places and to depths of 10 mm or more. It is interesting to note that, except for an area about 19 mm wide paralleling the channel, the erosion of the sample surface was no more severe than the surface pitting seen on the water-corrected sample.

**3. Chemical reactivity with oil additives.** Samples of the portland cured cement grout were immersed in saturated solutions of each of the additives used in the hydrostatic bearing oil and held at elevated (70°C) temperatures for about 200 hours to detect any deleterious action by these chemicals. While some specimens did show a small weight loss, (discussed later) no significant effect on the compressive strength of the specimens was seen. Chemical damage by the additives is not believed to be a factor in the grout failure.

The test specimens, 12.7-mm cubes cut from a grout sample obtained from the DSS 14 64-m antenna, are shown in Figs. 7 and 8. The control before (Fig. 8a) and after exposure (Fig. 8b) to the bearing oil shows virtually no change. Similarly, no effect on structure, weight or strength was observed with the Lubrizol (Fig. 7b) or PC-1244 foam suppressent samples.

The diphenylamine exposed sample (Fig. 8b) did exhibit some fractures in large aggregates at the surface. If real, this effect was probably caused by crystal growth (the samples were in a saturated solution in a water bath that cycled at ±4°C). The conditions of cycling temperature and particularly of saturation by the additive could not occur at the site and are therefore not believed to be a factor in the grout deterioration.

The sample exposed to 2,6-di-tert-butyl paracrysol (Fig. 7c) shows no evidence of concrete erosion but did lose 0.85% of its original weight during exposure. No effect on the compressive strength of the specimen was seen.

## C. Polymer Concrete Grouts

The two types of polymer concrete grouts proposed—the porous, "self-draining" open network and the void-free, solid grouts—differ widely in structure and in the properties needed in the matrix resin system. For example, in the open network structure, load distribution will be largely by particle-to-particle contact. Here, the modulus of the resin will not be as critical as with the solid composite grout. But the resin mass will be less and will require greater reactivity or a higher concentration of the curing agent. Eight epoxy resins and curing agents have been identified that, as primary resin systems or as diluents and modifiers, have the properties believed necessary in each of the two grout types. The resin systems and the test results obtained are described below.

**1. Epoxy resin selection.** Eight epoxy resins and curing agents were selected that, on the basis of cited literature values and manufacturers' data, had the properties considered necessary either as primary resin systems or diluents and modifiers for each of the two grout designs. The resins, listed in Table 1, are reaction products of epichlorohydrin and bisphenol-A, and differ mainly in the type of reactive diluents (such as butyl glycidyl ether and phenyl glycidyl ether) used to modify the viscosity of the system. Two exceptions, Epon 812 and 815, are low molecular weight aliphatic epoxies used to blend with and modify the viscosity, reactivity, and cure rate of the selected structural resins.

Triethylenetetramine (TETA) was used to cure all the resins because it represented the midpoint of the range of curing agents thought to be suitable for this application, based on the projected rate of cure, the reaction heat generated, and the degree of crosslinking obtained.

However, it became apparent in the preparation of the test cylinders that, because the grout was highly filled and the aggregate filler was an effective heat sink, the heat of polymerization of a TETA crosslinked system was not excessive. It now seems probable that faster curing agents, such as diethylenetriamine (DETA), may be used as a component of the curing agent.

**2. Compression strengths and modulus tests.** Replicate 7.62-cm-diameter by 15.25-cm-long compressive test cylinders containing 11.25% and 15.5% of each of the polymers were prepared with aggregate filler to approximate the structures of an open, self-draining system and a dense, void-free grout.

The cylinders were capped with a sulful/fly ash composition to assure uniform load application, and tested for compressive strength and modulus. Data for these tests are shown in Table 2.

The cylinders, shown under test in Fig. 9, typically failed catastrophically in a 45-deg shear mode, very similar to a dense, high-strength concrete, but at much higher loadings.

The greatest strength and modulus was obtained with Epon 828, a resin containing no reactive diluents. Generally, throughout the series, reactive diluents tended to lower the physical properties in proportion to their volume content. Thus, the 8132 resin specimens were lower in strength than the 828 resin specimens and, with the Ciba resins, the 509 resin system was lower in strength than the less diluted 507 system. The significance of these data is that all the epoxy resin grouts achieved very high strengths with moduli of near or greater than $1.38 \times 10^{10}$ N/m$^2$.

The aggregates used in these specimens were obtained from the Jet Propulsion Laboratory. They were screened to remove particles 6.4 mm in diameter and larger. For the porous grouts, particles finer than 1.5 mm were also removed to obtain the open structure and low resin content needed.

One mix was prepared to examine the properties when a more continuous size distribution was used. In this case only the fine particles smaller than 0.51 mm were removed. The resin content (15.5% Epon 828) and mixing and placement procedures were unchanged. The compressive strength obtained, $10.24 \times 10^7$ N/m$^2$, was the highest seen in this series, and the modulus $1.58 \times 10^{13}$ N/m$^2$ was significantly higher than any other in the series.

It now seems likely that with the proper design of the aggregate particle size distribution, the compressive strengths can be further increased and moduli of $2.07 \times 10^{13}$ N/m$^2$ or higher may be achieved.

**3. Erosion test.** The erosion test fixture previously described was also used to test the candidate polymer grouts.

The compression chamber sleeves bonded to the upper cylinder (Fig. 9) were machined to fit the piston to close tolerances (in most cases 0.05 to 0.13 mm on diameter). Within these tolerances, the oil flow between the sleeve and the piston was small compared to that flowing across the sample surface and out the exit port.

The test cycle selected was of six seconds duration (loading and unloading of the test specimen) providing a capability of 14,400 cycles per 24-hour day. The peak load setting of $2 \times 10^9$ N/m$^2$ represents a balance of relatively high stress (three times the actual load under the antenna legs) and short cycle times. Each piston stroke (6.35 mm) moved approximately 29.5 cm$^3$ of oil across the sample surface and out the 1.6-mm exhaust port in about 3 seconds, the high-pressure portion of the cycle.

The test apparatus and procedure for installing and adjusting the erosion cylinder are shown in Figs. 10 through 14. The test cylinder shown, a portland cement dry-pack specimen, is dimensionally identical to the polymer concrete cylinders.

**4. Polymer concrete erosion test results.** The polymer concrete grout samples, solid and porous, were exposed to severe erosion testing. No damage to either material was seen after more than 27,000 cycles (see Fig. 14). Under 10X magnification, fine machining marks transferred from the metal mold were still visible on the exposed surface.

Evidence that the porous grout would function to drain away trapped oil and eliminate the erosive effect was seen during the testing of these specimens. With the solid grout, the hydraulic pressure developed in the compression stroke was high enough that the exit port could not be closed by hand pressure. The oil flow was strong and continuous through the full downward piston stroke. By contrast, the exhaust port was easily closed by light finger pressure during testing of the porous sample. Here, a slight buildup of pressure could be felt, but the flow through the exit was readily stopped and the total volume of pumped oil moved easily through the sample.

**5. Creep characteristics of epoxy polymer grouts.** The creep characteristics of an epoxy polymer grout test cylinder were determined.

The test sample had the following composition:

| Component | Weight, g | % |
|---|---|---|
| Screened JPL aggregate (< 5-mm screen) | 2,704.0 | 84.50 |
| Epoxy resin (DOW DER 331) | 446.8 | 13.96 |
| Triethylenetetramine (TETA) | 49.1 | 1.53 |
| Totals | 3,199.9 | 99.99 |

DOW DER 331 epoxy is equivalent to Epon 828 which was used in this program. The grout test cylinder was 7.3 cm in diameter and 32.39 cm in length.

The creep for the epoxy grout was $3.5 \times 10^{-3}$ mm/mm under a 1.65-N/m$^2$ load while the typical total creep for portland cement concretes is much greater. Moreover, the creep data for the epoxy grout appears to have reached a fairly constant value of a creep shortening strain of $3.3 \times 10^{-3}$ to $4 \times 10^{-3}$ mm over a period of 23 days. This may be a limiting value for the creep to be expected for epoxy polymer grouts, again less than that of typical concrete (see Fig. 15).

The preceding data for the epoxy grout should be treated with caution as it is based on a single test sample, which had ample time to develop its full strength before a constant compressive load of 1.65 $N/m^2$ was applied. By contrast, the concrete creep tests cited were started seven days after the specimens are made and have a lower ($4.1 \times 10^6$ $N/m^2$) compressive load applied to them. Because of these considerations, the results of the creep test on epoxy grout are not directly comparable with those for concrete.

## D. Phase II: Developing, Refining, Optimizing of Selected Material

The work was planned for completion in two phases. Phase I was directed toward selecting five generic types of materials believed to be potentially able to meet these needs. Tests of physical properties, strength, modulus, resistance to erosion, and chemical attack by the oil were evaluated, and any unique requirements imposed by each material for mixing, placing, compacting, cooling (where high heat of polymerization might be a factor) were to be identified. The planned objective of Phase I was to identify the single generic type of material best suited to replace the dry pack grout. Phase II was to consist of developing, refining, and optimizing the selected material to achieve a high-strength, durable grout replacement.

The work of Phase I pointed up two potential routes to achieve the program goal. First, polymer grouts, with significantly higher strength, shorter cure time to reach load-bearing strength, and greater resistance to damage by leaking hydraulic oil, were promising replacement materials. Secondly, three possible factors were identified (namely early exposure to hydraulic oil, and the long cure time requirement), which in combination may contribute to premature failure of the portland cement grout. These deficiencies appeared to be correctable by small changes in the placement procedure and the use of a plastic film seal to prevent oil from entering a freshly repaired zone. The development and application of such procedures may make the dry-pack grout now used a completely acceptable material.

Each of these systems offers unique advantages as well as disadvantages, some of which were discovered only during their adaptation to this application. Phase II was designed to concurrently develop, test, and evaluate both systems, looking particularly at the large volume heating (polymer exotherm), curing rate, dimensional change, and oil resistance characteristics of each.

**1. Resin selection.** In the work statement developed for Phase I of the program, it was proposed that several epoxy resins with various viscosities be evaluated. In a given series of diglycidylethers of bisphenol A resins, as exemplified by Epon 828, lower viscosities are usually achieved by adding reactive diluents that are generally monofunctional. As such, they detract from the maximum physical properties attainable by the cured resin. The diluents generally make mixing and handling of the resins in preparing glass fabric reinforced structural laminates easier. However, it has been found that satisfactory mixes of Epon 828, alone and with aggregate, can be made despite the high viscosity of the pure resin. Based on these considerations, it was decided not to use reactive diluents. The use of such additives was proven unnecessary.

Therefore, all work on epoxy grouts during Phase II was done using Shell Chemical Co. Epon 828 which is a light-colored epichlorohydrin/bisphenol-A type low molecular weight epoxy resin. It is widely used in surface coating, laminating, casting, and potting applications. It has the following properties:

| Color 25°C (Gardner) (ASTM D1554-58T) | Viscosity 25°C, N · $S/m^2$ (ASTM D445-53T) | Epoxide equivalent[a] (ASTM D1652-59T) |
|---|---|---|
| 4 max | 10 to 16 | 185 to 192 |

[a]Epoxide equivalent = grams of resin containing 1-gram equivalent of epoxide

**2. Amine curing agent selection.** The primary objective of this task was to balance the catalyst type and concentration for the highest possible cure rate while maintaining a limited, acceptable heat of polymerization. Temperature and dimensional change (thermal expansion) of grouts were measured as functions of the catalyst type and concentration used.

Three amine curing agents were used. They were:

| Curing agent | Abbreviation | Equivalent weight, g |
|---|---|---|
| Ethylenediamine | (EDA) | 15.0 |
| Diethylenetriamine | (DETA) | 20.6 |
| Triethylenetetramine | (TETA) | 24.3 |

**3. Preliminary experiments.** The first experiments on these materials were performed on relatively small samples (5-cm cubes and cylinders of 7-cm diameter fitted with thermocouples). The purpose of these experiments was to determine

the rate at which strength developed during cure and the temperature (exotherm) attained during cure.

The compression test samples and exotherm samples were both prepared from single batches of resin. The time between the start of loading the first compression mold in a set to the completion of loading the cylindrical mold was in excess of an hour. The results of these experiments were inconclusive, since very small exotherms were observed and strength developed slowly. Based on experience, these results were not what would have been expected from an epoxy system. Accordingly, a study was made in which the mass of the resin/aggregate was increased until more consistent results were obtained.

**4. Basic curing properties of epoxy systems.** The first experiments in this series were performed using neat resin (no aggregate) and resin/aggregate mixtures packed loosely in plastic beakers. The purpose of these experiments was to furnish a base set of data for comparing further experiments. These results are summarized in Table 3.

These experiments were informative. They showed that extremely high exotherms could be anticipated with neat resin. The increased evidence of thermal degradation (darker color) with DETA and TETA even at lower maximum exotherms was noteworthy. One explanation for this was that with EDA and DETA increasing amounts of the heat of reaction were used in thermally degrading the polymer (darker color than with EDA). If this is so, the total heat of reaction could have been higher than was indicated by the maximum observed temperature. A second possible explanation could be that at higher temperature the DETA and TETA oxidize more readily than does EDA. Such oxidation could account for the observed dark color.

The moderating effect of the presence of 84.5% aggregate was evident in the lower maximum temperatures reached and the increase in the time to reach the maximum exotherm.

In this work, it was observed that ethylenediamine (EDA) fumed and gave off white vapors. This would be an undesirable characteristic in a repair system. Therefore, EDA was eliminated as a possible curing agent for polymer grout.

DETA proved to be an excellent curing agent for Epon 828, but exhibited a slightly higher exotherm than does TETA. Therefore, TETA was selected as the curing agent for the polymer grout in all subsequent work.

**5. Aggregate selection.** The bulk of the work was done using aggregate supplied by Jet Propulsion Laboratory. This was done as such aggregate is used presently at the antenna and a great deal of testing had gone into its selection for use in portland cement grouts. It was used both as received and after the large material (> 5-mm screen) had been removed.

**6. Curing studies.**

*a. Effect of quantity of grout on curing characteristics.* This portion of the work was directed toward determining the effect of mold size (weight of grout) on the curing characteristics. The Epon 828/triethylenetetramine (TETA) system was used in the work. Three different sizes and types of molds were used for these experiments. Relevant data on these molds are summarized as follows:

| Mold | Type | Weight of metal mass per unit volume of molding, g/cm$^3$ (g/in.$^3$) | |
|---|---|---|---|
| No. 1 | 3-cavity brass mold | 3.88 | (63.6) |
| No. 2 | Single-cavity, heavy-walled steel mold | 5.02 | (82.4) |
| No. 3 | Thin-walled steel mold | 0.09 | ( 1.4) |

Expansion during cure was observed with only the largest sample (4,680 g). Here the maximum expansion was 0.93% based on the 11.11-cm dimension. After cooling, the permanent expansion was 0.66%. This expansion occurred on an unrestrained grout surface.

The results of molding experiments using these molds are presented in Table 4. This work confirmed that the epoxy/grout system was sensitive to the mass and volume of the material present. As the mass of polymer increased, the rate of cure and the maximum temperature reached increased.

*b. Effect of oil on curing of Epon 828.* The cavities in the brass, three-cavity concrete mold (Mold 1) are 5.08 cm (2 in.) in diameter by 10.16 cm (4 in.) high [206 cm$^3$ (12.57 in.$^3$) or 0.002 m$^3$ (0.007 ft$^3$)]. The mold weighs 2,318 g. The three cavities are arranged side by side and three moldings were made at a time.

Cavity 1 was fitted with a thermocouple to measure the exotherm. Cavity 2 had a dial micrometer mounted over it. The foot of the micrometer was bearing on a plate positioned in the center of the top surface of the resin/sand mix.

Cavity 3 had hydraulic oil pooled on top of it to determine if oil had an adverse effect on the cure of the resin.

The presence of a pool of oil on the samples cured in the No. 1 mold did not appear to affect the surface cure of the

resin/aggregate mixture. The surfaces of samples cured with and without oil being present appeared to have the same degree of hardness (thus same degree of cure) when scratched by a steel probe.

*c. Effect of varying formulation and environment conditions on large-sample properties.* These samples were prepared in the thin metal molds (No. 3). The curing agent was varied (EDTA and EDA were substituted for TETA), additional TETA samples were cured with and without bottom insulation, and the proportion of resin to TETA and of aggregate to resin ratio was varied. These experiments are summarized in Table 5.

Figure 16 shows the apparatus used in these tests. A thermocouple was mounted in the approximate center of the aggregate/resin mass. The recorder monitored both the temperature at the center of the mass and the ambient air temperature. The mold was insulated on the sides with aluminum foil/glass wool insulation while the bottom was insulated (except in Run No. 4) with a 6.4-mm thick transite board. The entire assembly was set on a heavy steel plate, which was part of the support stand for a dial micrometer.

Changes in dimension along the longitudinal direction were measured using a dial micrometer. The foot of this micrometer was initially 0.013 mm from the 0.025-mm brass shim resting on the top of the aggregate/resin mass. Because of this, the micrometer's foot did not bear on the uncured surface. This prevented penetration of the foot into the grout during the early stages of the cure.

Strength development of the grout was measured using two soil test penetrometers which are normally used in measuring the bearing strength of soil.

The penetrometers could easily penetrate the mass of grout during most of the period during which the cure was taking place. However, the final stages of cure and the development of most of the strength occurred over a very brief interval of 2 to 3 minutes. For example, in Molding No. 1, the penetrometers could easily penetrate the grout mass at 65 to 67 minutes after curing agent was added to the epoxy. However, at 70 minutes the concrete penetrometer (maximum 4.83 $N/m^2$) would not even indent the surface under full load.

The maximum expansion occurs at the same time that sufficient strength develops to resist penetration. Both of these events precede the time at which the maximum exotherm occurs. The recorded expansions occurred on the unrestrained top surface of the grout. Subsequent work showed that the proper restraint of this surface expansion and contraction during cure ceased to be a problem.

This work indicated that mixes containing TETA cured more rapidly than those containing DETA or EDA. However, the maximum change in temperature was less with TETA. Expansion during cure was also smaller in the TETA cured grouts than in those using either DETA or EDA as curing agent.

The presence of an efficient heat sink (Molding 4) decreased the maximum temperature rise and increased the cure time, but did not appear to effect the strength properties of the final cured material.

Increasing the amount of aggregate in Run No. 5 also resulted in smaller temperature increases and slower cures. Again, the finished grout appeared to have the necessary strength properties. The results in Run No. 6 were anomalous. We would have expected an increased exotherm when the amount of aggregate was decreased (richer in resin). This did not happen. Similarly, we expected a somewhat more rapid reaction; instead it was actually slower. However, the quality of the cured grout appeared to be acceptable.

Decreasing the amount of curing agent had the expected effect of lowering the exotherm and increasing the reaction time.

Based on this work, using the Epon 828/TETA system, a useful working life of at least 65 minutes from the time amine was added to the resin could be expected. This was based on an 84.5/15.5 aggregate/resin mixture. This would indicate the possibility of increasing the working life of the grout by providing a more efficient heat sink, by adjustment of the resin/aggregate composition, or by adjustment of the ratio of epoxy resin to amine curing agent.

*d. Cure of warm epoxy grout mixtures.* Two experiments were made to determine the curing characteristics of epoxy grout mixtures in which all components were preheated to 38°C prior to the time they were mixed. The results were as follows:

| Parameter | Test 1 | Test 2 |
|---|---|---|
| Time to maximum temperature, min. | 55 | 52 |
| Maximum temperature, °C | 75 | 84 |
| Maximum ΔT, °C | | |
| Above temperature of grout components | 38 | 46 |
| Above ambient temperature | 55 | 63 |
| Condition of grout at time of maximum exotherm | Firm | Firm |

During Test 1, the epoxy grout was probed with a penetrometer to determine the time at which the material became unworkable and developed load bearing characteristics. This occurred about 50 minutes after the grout was mixed. The probing operation disturbed the dial micrometer that was measuring expansion, so we felt these readings were unreliable. We were also surprised that the maximum temperature was not higher than 75°C. Because of these factors this test was repeated (Test 2). The peak temperature was 84°C (9°C higher than in 1), but still only 11°C higher than tests started with all ingredients at 20 to 25°C. In Test 2, the grout was not probed and the total expansion was observed to be 0.35 mm or 0.3% of the height of the sample.

*e. Cure of cold epoxy grout mixtures.* It was of interest to determine the curing characteristics of epoxy grout when the resin, curing agent, and aggregate were chilled to 4°C prior to mixing. One experiment was attempted to determine this property. However, when the ingredients were cooled to the desired temperature, the epoxy resin was too viscous to be mixed with the aggregate. As mixing proceeded, the first aggregate wetted with resin formed a ball of material that could not be mixed with the remaining aggregate until the mass of material had warmed to approximately 13 to 16°C (55 to 60°F).

No further work was attempted on studying the low-temperature curing characteristics of the epoxy grout system. However, if it should be necessary to perform repair work during cold weather, it would be necessary to protect the raw materials (resin, curing agent, and aggregate) from very low temperatures by keeping them in a heated 15.5°C (60°F) building.

*f. Solution to the sagging properties of epoxy grout.* Previous experience by the Jet Propulsion Laboratory with epoxy grouts had indicated that they sag away from the top of the mold. This is an intolerable condition for the antenna leveling application. Because of this input from JPL it became imperative to determine whether our proposed epoxy grout would sag when used and if it did whether corrective measures could be taken to make the grout nonsagging.

*g. Determining the sagging characteristics of epoxy grouts.* Several experiments were performed to determine whether sagging would be a problem with the developed Epon 828/amine/aggregate grout. The first such experiment was performed by packing the 84.5% aggregate, 15.5% resin grout into a rigid wooden box that was open on one end. The epoxy grout was packed into the box from the end. During the packing operation it was observed that applying a load to one part of the grout caused it to flow and deform. Packing pressure on the top or bottom position of the grout caused it to extrude from the end of the box at a point away from the stress. After packing was complete, no slumping of the angle of repose of the grout was observed at the open end. However, examination of the grout adjacent to the top of the box revealed that there was approximately a 3.2-mm gap between the top of the grout and the top of the box. Additional tamping did not correct this deficiency.

Two approaches appeared possible ways of correcting this problem. They were:

(1) Addition of a thickening agent to the presently developed epoxy grout (84.5% aggregate, 15.5% resin).

(2) Reduction of the resin content of the grout to make a drier less fluid mix.

Both of these approaches to preventing sagging were tried, both alone and in combination with each other.

As the use of thickening agents, such as finely divided silica, was more straightforward, it was tried first. The following formulation was used in our initial experiments:

| Ingredient | Weight, g | % |
|---|---|---|
| Epon 828 | 273.9 | 13.4 |
| Triethylenetetramine | 36.1 | 1.8 |
| Sand (< 5-mm screen) | 1,690 | 82.8 |
| Finely divided silica | 40 | 2.0 |

This material was mixed and then packed into a steel mold that had a transparent (plexiglas) top. The mix appeared dry and when tamped did not have the fluid properties observed in grouts that did not contain finely divided silica.

There was no evidence of sagging when the material was viewed through the transparent top or from the open end (Fig. 7). The grout was removed from the mold after it had cured, but was still warm. The definite overhang at the top of the material and the ability of the tamped material to retain the impression of the tamper is noteworthy. Figure 8 shows the grout. The straight edge showed the sample was level across the top with no evidence of sagging.

This experiment was repeated but the epoxy grout was left in the mold until the material had cooled to room temperature. There was no visible evidence of sagging of either the warm material or after it had cooled. A piece of 0.025-mm brass shim stock could not be inserted between the top of the grout and the bottom of the plexiglas cover plate.

These initial experiments were encouraging and additional work was done on thickening agents and adjustments of the ratio of epoxy resin to aggregate.

*h. Development of epoxy grouts containing antisagging additives.* The initial experiments using a thickener (antisag additive) were encouraging. Thus, a series of experiments were undertaken to determine the effect of other thickeners on the sagging properties and compressive strengths of epoxy grouts.

The materials for these tests were hand mixed and packed into a steel mold fitted with a plexiglas top (Figs. 17 and 18) to determine sagging properties and a steel mold to produce 2.5-cm cubes for compression tests. The grouts were observed for evidence of sagging away from the top of the mold and compressive strength tests were made one week after the samples were prepared.

The results of this work is summarized in Table 6 and described as follows:

(1) All epoxy grouts that do not contain a thickening material sag when packed into the test mold.

(2) The strength of the grout decreases as the amount of thickening agent is increased.

(3) Between 1-1/2 and 2% thickening agent is needed to produce a nonsagging grout.

(4) Lowered resin content (< 15.5%) usually produces a grout with a lower compressive strength.

The samples made for determining compressive strength were 2.5-cm cubes and weighed an average of 36 g. The mold in which they were prepared weighed 8,123 g and contained three grout samples or 108 g of material. This gave a ratio of 75 g of metal per gram of grout material. These samples cured slowly in the presence of this large mass of metal and could be demolded in one day, at which time they were quite strong. This shows that a relatively small mass of epoxy grout will cure in the presence of a large heat sink.

*i. Filling intermediate-sized cavities with epoxy grout.* This work was undertaken to determine if an epoxy grout containing finely divided silica could be mixed in larger quantities and placed in a cavity without sagging.

7. **Application of epoxy grout to a 0.028-$m^3$ cavity.** This work was done using the following material:

| Ingredient | Weight, g |
|---|---|
| Aggregate (< 9.5-mm screen) | 17,760 |
| Finely divided silica | 320 |
| Epoxy (Epon 828) | 3,330 |

This formulation contained 1.5% silica thickener, which represented what was believed to be a minimum amount of thickener to prevent sagging. No curing agent was added to the epoxy. This permitted working with the grout without having to be concerned with the material curing in the mixer or while being placed in the cavity.

The cavity or mold was 45.7 cm long by 22.8 cm wide by 15.25 cm high. The mold had wooden sides, back and bottom. The front of the mold (22.8 cm × 15.25 cm) was left open and the top was closed with a plexiglas sheet that permitted observing the grout while it was being added and tamped. The mold had a volume of 15,900 $cm^3$.

A 141-$cm^3$ cement mixer was used in this experiment. The aggregate after screening out the large pebbles (> 9.5-mm screen) plus thickener was placed in the mixer and the opening was covered with a polyethylene film. These ingredients were mixed for two minutes and then the epoxy resin was added. Lumps of aggregate wetted with epoxy formed and rolled around the inner surface of the mixer. Continued mixing did not appear to be effective in breaking up these lumps. The mixer was stopped and the lumped material broken up using a large spatula. When mixing resumed, a reasonably uniform mix resulted.

This mixture was dumped out onto a plastic sheet and a portion of the "grout" was shoveled into the 22.8-cm × 45.7-cm × 15.25-cm cavity and the loose grout was tamped in place using a Skil hammer drill which delivered 3,750 blows per minute. The hammer drill was fitted with an adapter, extension arm, and a 5-cm × 5-cm foot. The mold was filled by alternate additions of loose grout and tamping. This grout was nonsagging across the 228-cm width of the cavity.

The grout was removed from the cavity and placed in the cement mixer and the necessary amount of amine (TETA) curing agent (52 g) added. The mixer was started and the amine mixed into the grout. Difficulty was once more experienced with the failure of the mixer to break up the lumps. The grout was transferred to a 9.5-liter plastic bucket and allowed to cure. A maximum exotherm of 67°C was obtained. The high exotherm probably reflected the poor heat transfer properties of the plastic bucket.

Compressive strength samples were also prepared from the grout. These samples had a very low strength value, namely 5.5 $N/m^2$. This low strength was attributed to the difficulty experienced in mixing.

It was concluded from this work that a cement mixer did not produce the type of mixing necessary for use in blending polymer grouts.

**8. Preparation of grouts using a mortar mixer.** To improve the mixing, a gasoline engine powered 0.2-$m^3$ mortar mixer was rented. This machine was used in all subsequent intermediate and full-scale grout tests.

The following formulation was used for this work:

| Ingredient | Weight, g |
|---|---|
| Aggregate (< 9.5-mm screen) | 17,760 |
| Finely divided silica thickener | 430 |
| Epon 828/TETA (3,050 g/401 g) | 3,330 |

The grout made from this formulation contained 2% thickener.

The aggregate and thickener were transferred to the mixer, which was closed with a polyethylene film. The dry ingredients were mixed for 5 minutes. During this time, the epoxy and amine curing agent were mixed for 2 minutes using a laboratory stirrer. This mixture was added to the aggregate and mixing continued for an additional 5 minutes.

At the end of mixing, the grout was discharged onto a plastic sheet. The grout was then transferred in small portions into an 48-cm X 48-cm X 15-cm cavity and then tamped in place using a hammer drill equipped with an extension rod and a 7.6-cm square foot. This continued until all grout from the mix was in place. Figure 10 shows the mold. Figure 20 shows the filling of this cavity, and Fig. 21 shows the partially filled cavity.

Immediately after placing the first batch, a second similar batch was made up and placed in the cavity using a similar emplacement procedure with the exception that mixing time was reduced from 5 to 2 minutes.

**9. Use of epoxy grout to fill a 0.08-$m^3$ cavity.** At the end of the Phase II program, the epoxy grout formula that was developed as a result of this work was used to fill a 0.08-$m^3$ cavity (see Fig. 19). The filling techniques were modeled on the methods used by JPL to emplace the present portland cement grout system, and adapted by MRC for use with epoxy grout.

A test site for the demonstration of the use of epoxy grout was built on an existing concrete slab. It is shown in Fig. 22. It provided a 46-cm X 112-cm X 15-cm cavity that approximates the size of the grout replacement section at Goldstone. One end of the cavity was closed with a 1.27-cm-thick steel plate that provided a bucking plate against which the grout could be compacted. The top was covered with 2.54-cm-thick steel plates.

Figure 13 also shows the details of the method adopted to hold down the top plates. During the emplacement and curing of the grout, it was learned that the method was not entirely satisfactory. The thermal expansion of the grout caused a very slight lifting or bowing of the angle iron. As a consequence, more deflection of the steel cover plates was observed adjacent to the hold downs than was observed along the center line of the plates.

**10. Composition of epoxy grout used in filling a large cavity.** Two 86.26-kg batches were used to fill the 45.7-cm X 112-cm X 15-cm cavity. Each batch had the following composition:

| Ingredient | Weight, g | % | Source |
|---|---|---|---|
| Aggregate, < 9.5-mm screen | 71,070 | 82.07 | JPL |
| Thickener | 1,720 | 1.99 | Cabot Corp. |
| Epon 828 | 12,200 | 14.09 | Shell Chemical |
| Triethylenetetramine (TETA) | 1,600 | 1.85 | Dow Chemical |
| Total | 86,590 | 100.00 | |

The above formulation has the following composition:

| Ingredient | % |
|---|---|
| Filler (aggregate + Cab-O-Sil) | 84.06 |
| Resin (epoxy + TETA) | 15.94 |

This formulation is apparently a half percent richer in epoxy than the formulations used in the intermediate size tests. The

reason for this apparent discrepancy is that the epoxy resin and curing agent (TETA) was mixed in four separate batches, each having the composition:

| Ingredient | Weight, g |
|---|---|
| Epoxy 828 | 3050 |
| TETA | 401 |

Mixing was done in four 3.785-liter paint cans. This operation is shown in Fig. 23.

When mixing was complete, the epoxy resin was poured from the can to the mortar mixer. In this operation, retention of resin in the can amounted to about 84 g/can. With this retention of about 336 g (84 g × 4 cans), the actual composition of the grout as emplaced follows:

| Ingredient | Weight, g | % |
|---|---|---|
| JPL aggregate (< 9.5-mm screen) | 71,070 | 82.39 |
| Thickener | 1,720 | 2.00 |
| Epoxy + TETA | 13,470 | 15.61 |
| Total | 86,260 | 100.00 |

This represents a charge of 86.26 kg that was actually mixed. The percentages are quite close to the intended composition of 84.5% aggregate and 15.5% resin. The small excess of resin would not be sufficient to significantly affect the properties of the grout.

**11. Placement of epoxy grout in test cavity.** The two 86.26-kg batches of epoxy grout material were mixed in a 0.2-$m^3$ mortar mixer driven by an 6-kW gasoline engine. The engine drove the paddles of the mixer through a V-belt. This test was done early in the day so that the test site was not heated by direct sunlight. It was felt such heating would have accelerated the cure of the grout.

Each batch was mixed separately in the mixer, and the second batch was mixed immediately after the first batch was dumped.

The log for the mixing of the grout and its emplacement and tamping in the test cavity was kept. The most notable thing about the filling of this cavity was that all the epoxy grout was mixed and tamped into place in 59 minutes from the start of the test and the last placed material had a bearing strength in excess of $4.8 \times 10^6$ $N/m^2$ 1 hour and 32 minutes after the start of the test.

Note oil was injected into the top of the cavity to simulate the presence of oil in some regional areas at Goldstone. Special attention was given to the cure and strength of these areas to determine detrimental effects from the oil if any. See Fig. 25.

The areas of grout surrounding the holes through which oil was introduced during the placement and tamping were quite strong and did not appear to have been effected by the oil. These areas are as resistant to scratching by a steel probe as are the areas some distance from the oil.

**12. Thermal effects of use of epoxy grout in a large cavity.** When epoxy is cured by an amine there is a fairly large exotherm (about 25 kcal/mole of epoxy (Ref. 1). This exotherm can cause straight resins to reach quite high temperatures when they cure. However, in the grout system the large proportion of aggregate present acts as a heat sink and results in considerably lower temperatures.

An examination of the grout after the top plates were removed did not reveal any evidence of thermal stresses and cracks in the surface of the grout. There was also no evidence of thermal degradation during the cure and after removal of the top plates.

**13. Dimensional stability of epoxy grout.** Dimensional stability of a large mass of epoxy grout is of considerable concern. Measurements, using a depth micrometer, of dimensional change during cure and immediately thereafter, and visual observation using a straight edge of the overall levelness of the top surface, were made after the removal of the top cover plates.

The expansion and/or contraction of the grout during cure was measured using a depth micrometer to measure the distance from an aluminum angle to the top of the steel cover plates. These measurements were made across the width of the cavity and 56 cm from the back plate. The details of how these measurements were made along with the results are given in Fig. 24. Each distance from the top of the horizontal leg of the angle to the top of the steel plate represent the average of three individual measurements.

Some difficulty was experienced in making these measurements as the aluminum angle had a tendency to bend slightly over the 70-cm space, if any substantial load was placed on it. Great care was taken not to press down on the depth micrometer during the making of a measurement. It is believed they

are reliable values, in view of the care taken in making them, and they are averages of three individual readings.

All of the stations showed a slight expansion of the grout at the maximum exotherm and consequent lifting of the cover plate. These displacements generally became larger as the east side of the cavity was approached. It is believed that this increase was due to a slight lifting or rotation of the angle iron hold down. This positive displacement, along the east side of the cavity, persisted even after the grout had cooled to ambient temperature.

Some possible slight contraction of 0.025 to 0.05 mm was observed at Stations B and C after the grout had cooled. However, it is believed that these were errors in reading, as the readings at the adjacent stations (A and D) are not consistent with shrinkage having occurred. The easy removal of the cover plates also was not consistent with strong enough adhesive forces between the grout and the steel to expect that the 2.54-cm steel plates would be pulled downward.

After the removal of the cover plates, the flatness of the top of the grout was checked using a steel straight edge, as shown in Fig. 24. It was found that the grout was essentially flat when checked across the width at various positions along the 112-cm length of the cavity. When a straight edge was laid across the grout, a uniform thin band of light was observed between the straight edge and the grout surface.

When the straight edge was laid along the 112-cm axis of the grout it was found that a 381-$\mu$m shim could be slipped under the straight edge and slipped back and forth along the length of the grout, except at the ends where the ends of the straight edge were in contact with the grout. This was a disturbing observation as it appeared that the top surface of the grout was somewhat concave in the long dimension of the cavity.

However, further examination of the test site revealed that the concrete sides of the cavity had a similar contour, namely higher at the ends than in the middle of the length. A steel straight edge laid on either the grout or the concrete revealed the same clearance between the straight edge and the surface under it. Apparently, the steel cover plates were merely following the contour of the concrete side pieces.

All the data indicates that in the presence of large heat sinks and relatively heavy steel top plates, the grout upon curing does not exhibit an excessive amount of expansion or contraction and, if such displacement occur, they are highly uniform.

**14. Compressive strength of epoxy grout used in prototype repair test.** Samples of the grout material from both 86-kg batches were taken and used to make compressive strength (2.5 $cm^3$). These samples were tested for compressive strength after they had aged for one week. The results are as follows:

| Batch | Compressive strength; average of 3 samples, $N/m^2$ | Standard deviation, $N/m^2$ |
|---|---|---|
| 1 | $1.7 \times 10^8$ | $\pm 4.34 \times 10^6$ |
| 2 | $1.57 \times 10^8$ | $4.9 \times 10^6$ |

The high compressive strengths of these samples can perhaps be attributed to the improved mixing achieved in the mortar mixer.

## E. Development of Sealing Films for Uncured Portland Cement Grouts

Work was done on developing a sealing film to protect portland cement grout from attack by oil during cure.

**1. Sprayed on thin-film barriers.** To accomplish this, an apparatus to test the sealing properties of the candidate film materials was designed and built. The test equipment is shown schematically in Fig. 25. Hydraulic oil under nitrogen pressure (0 to $6.9 \times 10^6$ $N/m^2$) was supplied to ports in the sides of a concrete mold. The port on the left side of the mold was 0.25 mm in diameter and provided direct access of the pressurized oil to the back of the film. On the right side of the test fixture, two steel plates were used to simulate the junction between sole plates and the resulting elongated opening.

In use, the inside surface of the mold was coated with the film former that was being tested. The oil supply lines were filled with oil prior to the start of the test. After the film had set, the mold cavity was filled with portland cement grout. Oil pressure was applied to the film/grout after various curing times.

To establish a control for the film experiments and to work out experimental details, portland cement grouts were tested. These grouts have the following formula:

| Ingredient | % |
|---|---|
| Aggregate (through 5-mm screen) | 55.3 |
| Portland cement | 36.3 |
| Water | 8.4 |

After mixing, the grouts were packed into the mold and allowed different times to cure, as shown in the following table. After these grout cure times, oil pressure was applied to the film/grout in $3.45 \times 10^5$ N/m$^2$ increments at 5-minute intervals.

| Time after addition of water to application of oil pressure, h | Results |
|---|---|
| 1 | Failed at $5.5 \times 10^6$ N/m$^2$ 2 hours and 15 minutes after water addition. Failure evidenced by oil flowing out of top of mold. |
| 2 | No failure to $5.9 \times 10^6$ N/m$^2$. Some slight pressure drop after each increment of pressure. |
| 4 | Same as at 2 hours. |

These tests indicate that after 2 hours cure the cement was sufficiently cured to withstand $5.86 \times 10^6$ N/m$^2$ pressure. Therefore, all tests on paint films and tapes were started one hour after water addition to the portland cement grout.

The film formers listed in Table 7 were tested using various methods of application and cure times for the films. Oil pressure was applied to the back side of the film one hour after water addition to the cement/aggregate mix.

Of these candidate film formers, the laboratory mixed epoxy, Sherwin-Williams Superfast Dri synthetic paint, and Dow Corning's silicone caulk did not fail below $4.14 \times 10^6$ N/m$^2$ oil pressure. This indicates they might perform satisfactorily. However, they must be cured 20 hours before use. This surpasses the 2- to 4-hour time limit allowed for repairs. This allowance is based on the permissible down time for an antenna.

**2. Preformed films as seals to prevent the entrance of oil.** A study was made using preformed films and tapes that have pressure sensitive adhesives on one surface. These tapes were placed over the inside surface of the test jig where leakage of oil might occur (0.025-mm hole and simulated joint between sole plates). The tape was pressed in place and the mold immediately packed with portland cement grout. This work is summarized in Table 8.

This work indicates the use of preformed films is a promising approach, but work remains to be done developing a satisfactory means of applying such film to the interior of a repair cavity.

## III. Conclusions

As a result of work done during this program it can be concluded that an epoxy grout containing minor amounts of thickening agents is a feasible system for the repair of existing antenna support structures. This conclusion is based on the following:

(1) The placement of 177 kg of epoxy grout in a simulated repair cavity (0.078 m$^3$). The methods of placement are quite similar to the techniques currently in use for portland cement grouts.

(2) The rapid cure and consequent early development of load bearing properties by the epoxy grout. The epoxy grout becomes load bearing in approximately 2 hours after placement of a large amount of grout.

(3) The ability of epoxy grout to cure in the presence of a large heat sink (surrounding antenna support structure).

(4) Strengths considerably in excess of those realized by portland cement grouts.

(5) Good resistance during cure to hydraulic oil.

(6) Good dimensional stability when restrained by the surrounding structure.

(7) Low creep values under constant compressive loads.

(8) Readily available commercial ingredients are used to make up the grout.

(9) Safe working and handling techniques for epoxy resins are well established in industry.

In addition to the epoxy system for repairing antenna structures, a system was developed that shows promise of providing a means of protecting uncured portland cement grout from the harmful effects of hydraulic oil. This system is based on the use of preformed plastic and metal tapes that have a pressure sensitive adhesive on one face. These tapes can adhere to metal and concrete to seal openings against the entry of oil.

# Reference

1. Lee, H., and Neville, K., *Handbook of Epoxy Resins*, p. 6-3. McGraw-Hill Book Co., New York, N.Y., 1967.

**Table 1. Candidate epoxy resins and curing agents**

| Resins | Epoxide eq. wt. | Viscosity, N • S/m$^2$ | Curing agents | Amine eq. wt. |
|---|---|---|---|---|
| Epon 812 | 188 | 0.4 | Triethylenetetramine[a] (TETA) | 24 |
| Epon 815 | 185 | 0.6 | Diethylenetriamine[a] (DETA) | 21 |
| Epon 820[a] | 188 | 7.0 | Epon V-25 | 163 |
| Epon 825 | 175 | 5.0 | Epon V-40 | 140 |
| Epon 826[a] | 182 | 8.0 | Epon V-50[a] | 130 |
| Epon 828[a] | 188 | 13.0 | Epon V | 48 |
| Ciba 507[a] | 189 | 0.6 | Ciba 837[a] | 34 |
| Ciba 509[a] | 194 | 0.6 | Ciba 2964 | (30) |

[a]Primary resin and curing agent candidates.

**Table 2. Compressive strengths and modulus of polymer concrete samples**

| Code | Polymer[a] | Polymer content, wt% | Grout type | Compression strength, N/m$^2$ x $10^7$ | MOE,[b] N/m$^2$ x $10^{10}$ |
|---|---|---|---|---|---|
| 207895-1 | 828 | 11.25 | Open | 7.10 | 1.24 |
| 207895-2 | 828 | 15.5 | Solid | 9.62 | 1.38 |
| 207898-1 | 507 | 11.25 | Open | 6.44 | 1.03 |
| 207895-3 | 507 | 15.5 | Solid | 8.46 | 1.24 |
| 207895-5 | 509 | 11.25 | Open | 5.61 | –[c] |
| 207895-4 | 509 | 15.5 | Solid | 6.61 | –[c] |
| 207898-2 | 8132 | 11.25 | Open | 5.49 | –[c] |
| 207898-3 | 8132 | 15.5 | Solid | 7.58 | –[c] |
| 207898-4 | 826 | 11.25 | Open | 5.98 | 1.1 |
| 207898-5 | 826 | 15.5 | Solid | 9.23 | 1.38 |
| 1213901 | 828 | 15.5 | Solid | 10.24 | 1.59 |

[a]Epoxy resin designation.

[b]Modulus of elasticity.

[c]Not available.

**Table 3. Basic curing data on Epon 828 resin systems**

| Resin | Curing agent | | Aggregate[a] | Mold diameter,[b] cm (in.) | Maximum temperature, °C | Maximum ΔT, °C | Time to maximum temperature,[c] min | Comment |
|---|---|---|---|---|---|---|---|---|
| | Type | Amount | | | | | | |
| 200 | EDA | 16 | –[d] | 7.0 (2¾) | 202 | 180 | 48 | In all cases, beaker melts degradation as evidenced by color, least severe with EDA and most severe with TETA. |
| 200 | DETA | 21.8 | –[d] | 7.0 (2¾) | 196 | 174 | 46 | |
| 200 | TETA | 25.8 | –[d] | 7.0 (2¾) | 190 | 168 | 49 | |
| 100 | EDA | 8 | 589 | 9.5 (3¾) | 54 | 30 | 68 | All material hard and firm when maximum exotherm is reached. |
| 100 | DETA | 11 | 604 | 9.5 (3¾) | 51 | 27 | 71 | |
| 100 | TETA | 13 | 615 | 9.5 (3¾) | 45 | 20 | 72 | |

[a]JPL aggregate screened through 5-mm screen.
[b]Plastic beakers – 400 ml for neat resin and 1,000 ml for resin/aggregate.
[c]Maximum time was measured from time curing agent was added to resin.
[d]Not applicable.

**Table 4. Effect of resin/aggregate mix volume and weight on curing characteristics**

| Mold No. | Type | Weight[a] of resin aggregate | Maximum temperature, °C | Maximum ΔT, °C | Time to[b] maximum ΔT, min | Comment |
|---|---|---|---|---|---|---|
| 1 | Brass 3-cavity | 430 g/cavity | 27.0 | 2.0 | 160 | Molds uninsulated – large heat losses to surroundings. Very low exotherm – samples well set up after 160 min. No observable expansion during cure. Oil on surface did not adversely effect cure of surface. |
| 1 | Brass 3-cavity | 430 g/cavity | 34.5 | 7.5 | 108 | Mold insulated bottom and sides – increased exotherm – samples well set up in 108 minutes. No observed expansion. Surface wet with oil well cured. |
| 2 | Thick-walled steel mold | 2,850 g | 46.0 | 20.0 | 99 | Mold insulated top and bottom. Exotherm increases. No observable expansion. |
| 3 | Thin-walled steel mold | 4,680 g | 73.0 | 47.0 | 80 | Mold insulated top and bottom. Increased exotherm. Total expansion 0.1 cm (0.0405 in.) at 70 minutes. Increase in length after cooling (0.07 cm) (0.029 in.). |

[a]Resin/curing agent mixture 88.6/11.4, stoichiometric amounts. Aggregate/resin mix 84.5/15.5.
[b]Time from addition of curing agent to resin.

**Table 5. The effect of formulation variations on the curing characteristics of 2,220-cm³ (134-in.³) aggregate/resin moldings[a]**

| Molding | Resin mix | | Type | Resin/aggregate | | Maximum temperature, °C | Maximum exotherm, °C | Time to maximum exotherm[b], min | Linear expansion | | Time until penetrometer does not penetrate grout, 4.83 N/m² min | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Per cent epoxy | Per-cent amine | | Per-cent resin | Percent aggregate | | | | Maximum percent at time t, min | Residual after cooling | | |
| 1 | 88.6 | 11.4 | TETA | 15.5 | 84.5 | 73 | 47 | 80 | 0.93 at 70 | 0.66% when cold | 70 | Stoichiometric resin/curing agent mix of Epon 828 and EDTA. Insulated sides and bottom. |
| 2 | 99.0 | 9.9 | EDTA | 15.5 | 84.5 | 77.5 | 52.5 | 85 | 1.9 at 70 | 1.6% when cold | 70 | Stoichiometric resin/curing agent mix of Epon 828 and EDTA. Insulated sides and bottom. |
| 3 | 92.6 | 7.4 | EDA | 15.5 | 84.5 | 74 | 53 | 107 | 2.0 at 92 | 1.1% when cold | 94 | Stoichiometric resin/curing agent mix of Epon 828 and EDA. Insulated sides and bottom. |
| 4 | 88.6 | 11.4 | TETA | 15.5 | 84.5 | 67 | 44 | 105 | 0.5 at 82 | 0.2% when cold | 82 | Stoichiometric amounts of Epon 828 and TETA. Mold insulated on sides. Bottom uninsulated, resting on heavy steel plate. |
| 5 | 88.6 | 11.4 | TETA | 14.2 | 85.8 | 61 | 40 | 100 | 0.1 at 79 | | 79 | Stoichiometric amounts of Epon 828 and TETA. Increased aggregate content in mix. Sides and bottom insulated. |
| 6 | 88.6 | 11.4 | TETA | 17 | 83 | 69 | 47 | 108 | 0.93 at 90 | 0.57% when cold | 93 | Stoichiometric amounts of Epon 828 and TETA. Mold insulated sides and bottom. Decreased aggregate content. |
| 7 | 89.6 | 10.4 | TETA | 15.5 | 84.5 | 65 | 42 | 115 | 0.8 at 92 | 0.5% when cold | 92 | Less than stoichiometric amount of amine. Aggregate resin mix 84.5/15.5. Insulated sides and bottom. |

[a]Moldings made in Mold No. 3.

[b]Time measured for time curing agent is added to resin.

**Table 6. Characteristics of grout formulations**

| No. | Type | Resin[a], % | Aggregate[b] | Thickener[d] | Compression strength[c], 70.3 kgf/cm$^2$, (kpsi) | Characteristics |
|---|---|---|---|---|---|---|
| 624 | Portland cement | Cement 36 | < 5-mm screen, 55% | –[e] | 860 (12.3) | Nonsag |
| 625 | Portland cement | Cement 36 | > 5-mm screen, 55% | –[e] | 630 (8.9) | Slight sag |
| 651 | Epoxy | 17.5 | < 9.5 mm | –[e] | 1,620 (23.0) | Sags |
| 636 | Epoxy | 15.5 | < 9.5 mm | –[e] | 1,880 (26.7) | Sags |
| 650 | Epoxy | 15.5 | < 5-mm screen | –[e] | 1,510 (21.5) | Sags |
| 627 | Epoxy | 12.0 | < 5-mm screen | –[e] | 1,650 (23.4) | Sags |
| 629 | Epoxy | 11.5 | < 5-mm screen | –[e] | 1,800 (25.6) | Sags |
| 628 | Epoxy | 11.5 | Not screened | –[e] | 560 (8.0) | Sags |
| 638 | Epoxy | 10.0 | < 5-mm screen | –[e] | 730 (10.4) | Sags |
| 635 | Epoxy | 8.0 | < 5-mm screen | –[e] | 710 (10.1) | Sags |
| 632 | Epoxy | 15.5 | < 5-mm screen | 2 | 1,470 (20.9) | Nonsag |
| 652 | Epoxy | 17.5 | < 9.5 mm | 3% | 1,330 (18.9) | Nonsag |
| 649 | Epoxy | 17.5 | < 9.5 mm | 4% | 1,370 (19.5) | Nonsag |
| 655[d] | Epoxy | 15.5 | < 4.7 mm, not dried | 1.0 | 1,620 (23.1) | Sags |
| 654 | Epoxy | 15.5 | < 5-mm screen | 1.0 | 1,480 (21.1) | Slight sag |
| 656 | Epoxy | 15.5 | < 5-mm screen, not dried | 1.5 | 1,500 (21.4) | Nonsag |
| 645 | Epoxy | 15.5 | 5-mm screen, not dried | 2.0 | 1,470 (20.9) | Nonsag |
| 647 | Epoxy | 15.5 | | 2.0 | 1,440 (20.5) | Nonsag |
| 653 | Epoxy | 15.5 | < 5-mm screen, not dried | 2.0 | 1,410 (20.0) | Nonsag |
| 648 | Epoxy | 15.5 | 9.5 mm | 3.0 | 1,360 (19.4) | Nonsag |
| 646 | Epoxy | 15.5 | < 5-mm screen | 4.0 | 740 (10.5) | Nonsag |
| 642 | Epoxy | 10.0 | < 5-mm screen | 2.0 | 710 (10.1) | Nonsag |
| 643 | Epoxy | 10.0 | < 5-mm screen | 4.0 | 530 (7.5) | Nonsag |

[a]Percent resin represents combined Epon 828 and amine curing agent.
[b]All aggregates are Jet Propulsion Laboratory material obtained from San Gabriel River drainage.
[c]All tests run on 2.54-cm cubes that were individually molded. Based on one sample; other samples damaged during demolding.
[d]Cab-O-Sil M-S mode by Cabot and Co.
[e]Not applicable.

**Table 7. Evaluation of film forming systems**

| Type | Manufacturer | Method of application | Time to cure film | Pressure at failure[a], $N/m^2$ | Cause of failure |
|---|---|---|---|---|---|
| Epoxy | Lab mix | Spray | 40 min. | $6.9 \times 10^5$ | Unknown. |
| Epoxy | Lab mix | Brush | 20 h | $4.1 \times 10^6$ | Unknown. |
| Urethane (Polane) | Sherwin-Williams | Spray | 20 h | – | Oil continued to seep through film. |
| Superfase Dri synthetic paint | Sherwin-Williams | Spray | 2 h | $4.8 \times 10^6$ | Unknown. |
| Traffic paint | Sherwin-Williams | Brush | – | – | Oil seeps through film. |
| Automotive undercoat | Sherwin-Williams | Sprayed | 20 h | $6.9 \times 10^5$ | Oil under pressure washed undercoat away. |
| Polyurethane wood varnish | Jewel Paint Co. | Spray 2X | 2 h each coat plus | $3.45 \times 10^5$ | Poor film strength. |
| Floor enamel | Pratt & Lambert | Brush | 4 h, plus 1 h in contact with concrete | $6.9 \times 10^5$ | –[b] |
| Floor enamel | Pratt & Lambert | Brush | 4 h, plus 21 h in contact with concrete | $2.75 \times 10^6$ | –[b] |
| Hydroflex swimming pool paint | Lox System, Inc. | Brush | 3 h, plus 1 h in contact with concrete | $6.9 \times 10^5$ | –[b] |
| Thickened epoxy | Lab | Brush | 3 h, plus 1 h in contact with concrete | $2.75 \times 10^6$ | Slight leak through film. |
| Silicone rubber caulk | Dow Corning | Spread on with knife | 20 h | $5.17 \times 10^6$ | Mold rusted, possibly caused by acetic acid liberated during cure. |

[a]Failure is considered to have taken place when pressure drops of $3.45 \times 10^5$ $N/m^2$ or more occur in less than a minute.
[b]Not applicable.

**Table 8. Use of preformed films as oil barriers**

| Type | Manufacturer | Pressure applied $N/m^2$ | Failure | Comment |
|---|---|---|---|---|
| Brown plastic tape 6487DK02-4 | Borden | $5.86 \times 10^6$ | Yes | Film damaged by tamping tool. |
| Aluminum foil | –[a] | $5.86 \times 10^6$ | No | –[a] |
| Electrical aluminum tape | 3M | $5.86 \times 10^6$ | No | –[a] |
| Trifoil type 603 | Oak Materials Group, Tripoint Division | $5.86 \times 10^6$ | No | –[a] |
| Teflon tape | 3M | $5.86 \times 10^6$ | Yes | Film damaged by tamping tool. |
| Lamotape 980 (aluminum on Mylar) | Lamotite | $5.86 \times 10^6$ | No | –[a] |

[a]Not applicable.

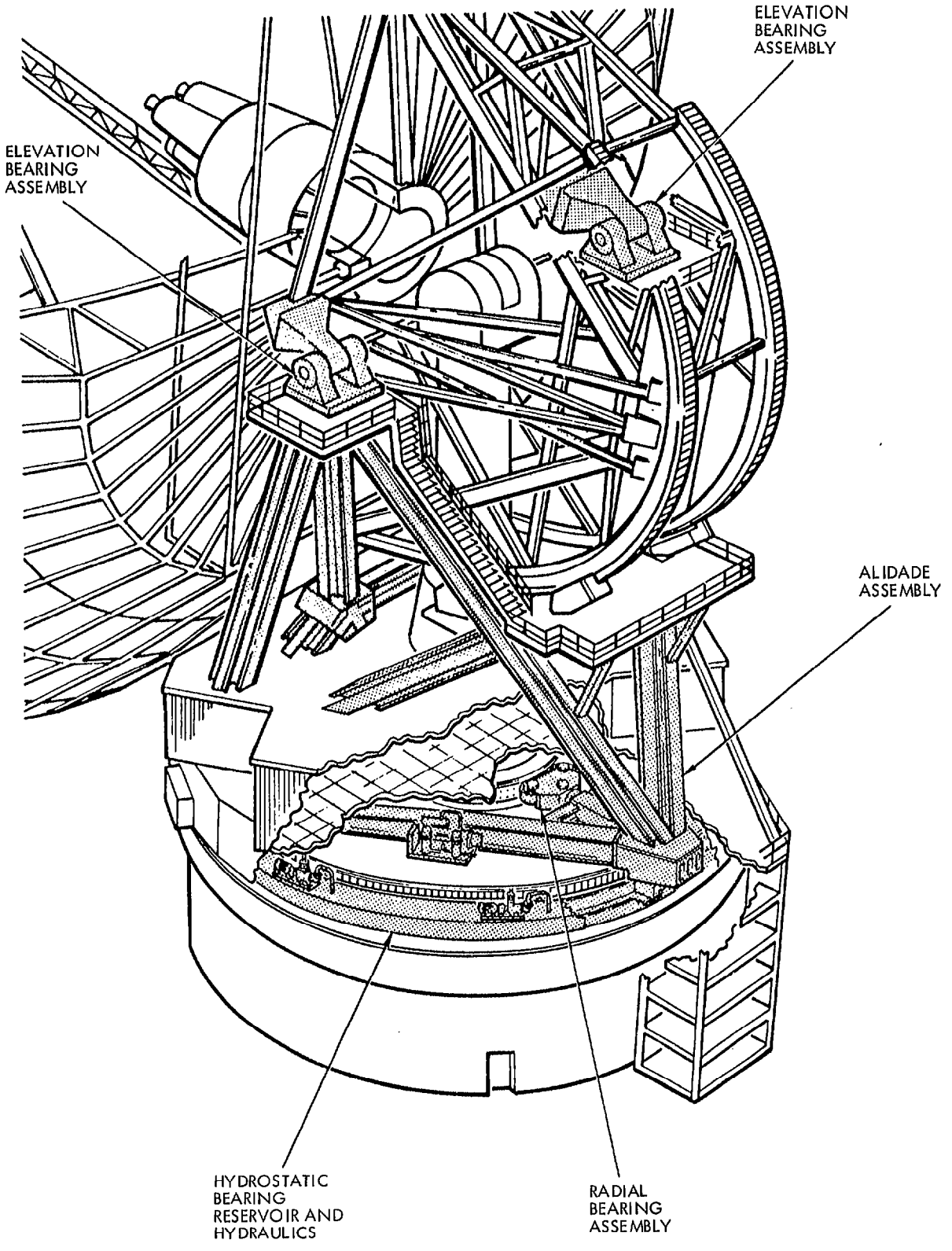



**Fig. 1. Antenna mount**

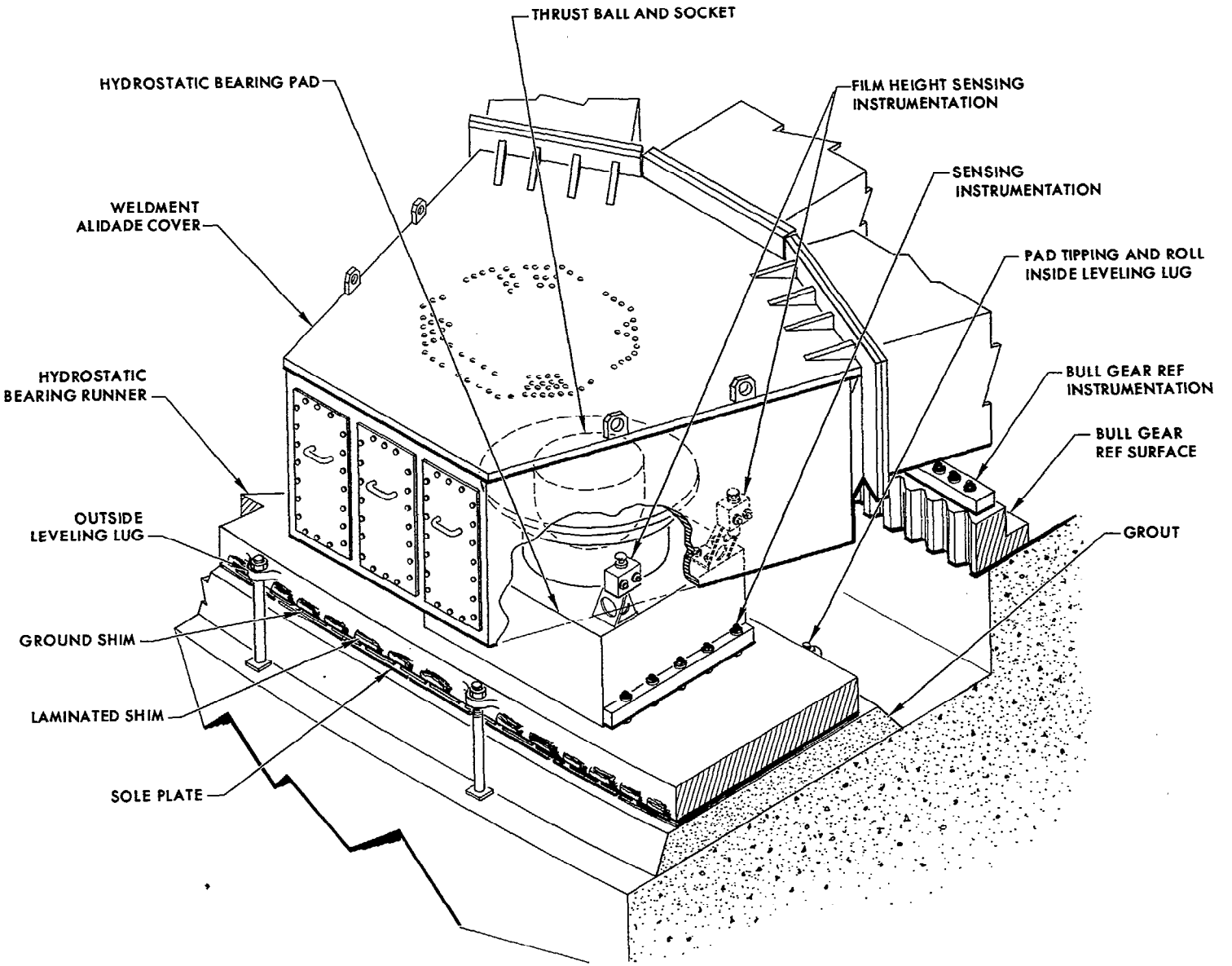
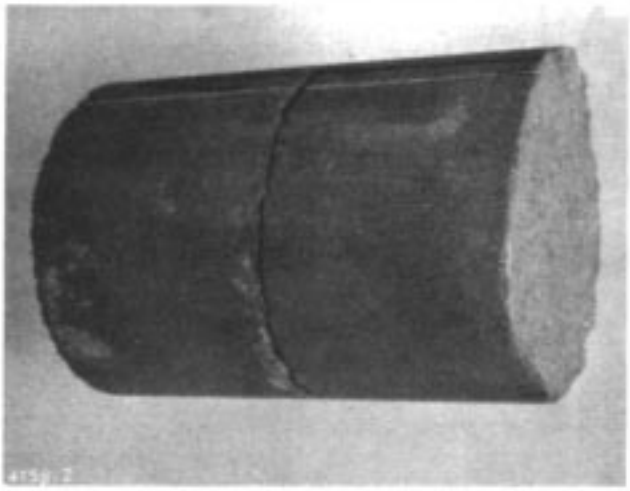
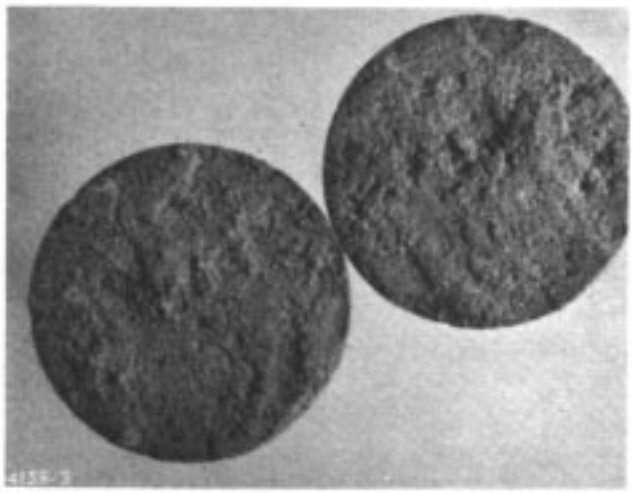


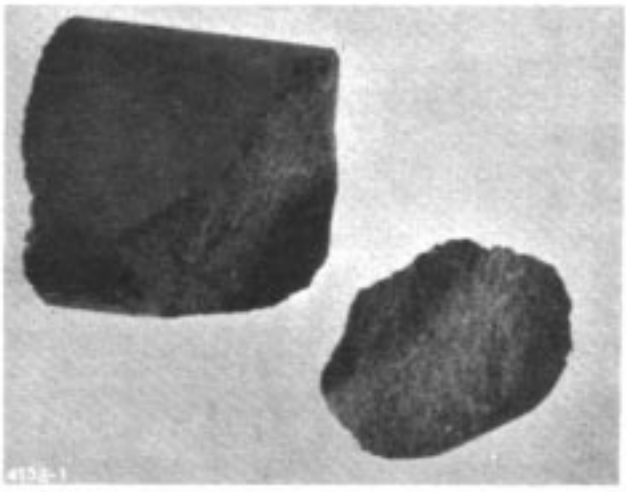

**Fig. 2. Hydrostatic bearing cross section**



(a) FRACTURES CONSISTENTLY OCCURRED ACROSS THE PACKING INTERFACE.

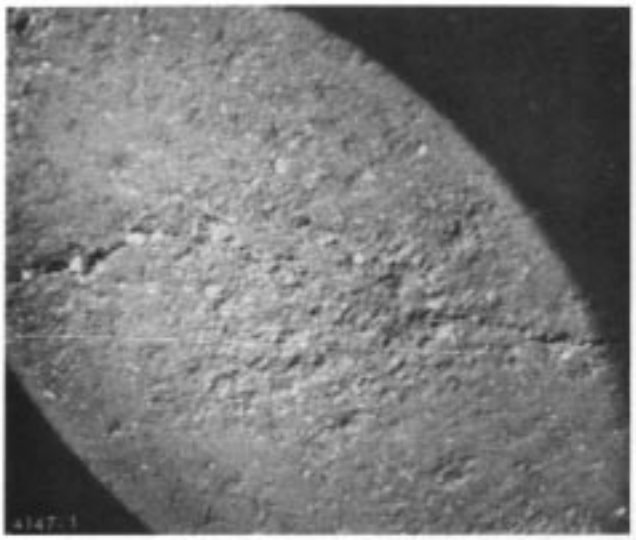


(b) PACKING RAM IMPRINTS ON THE FRACTURE SURFACE SHOW THE CYLINDER PARTED AT THE PACKING INTERFACE.

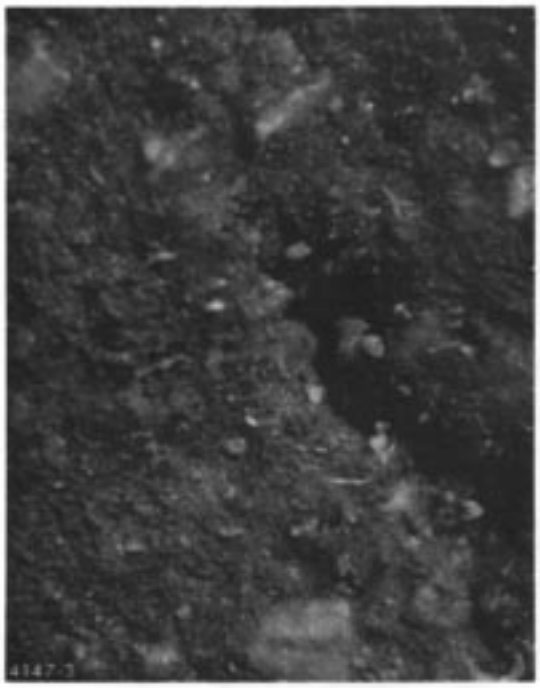


(c) AN IMPACT FRACTURE WITHIN A SINGLE PACKING INCREMENT FOLLOWS THE RANDOM PATH OF A SOUND CONCRETE.

**Fig. 3. The effects of water loss. The loss of 5% to 6% of the mix water caused dry, weakly bonded packing interfaces, but material within a single packing increment underwent normal cure**



(a) BROAD, SHALLOW EROSION AREAS ARE CONNECTED BY DEEPER CHANNELS.



(b) THE CHANNELS FORM A CONTINUOUS NETWORK OVER THE GROUT SURFACE.

**Fig. 4. The eroded areas and channels in a grout sample removed from the Goldstone site**

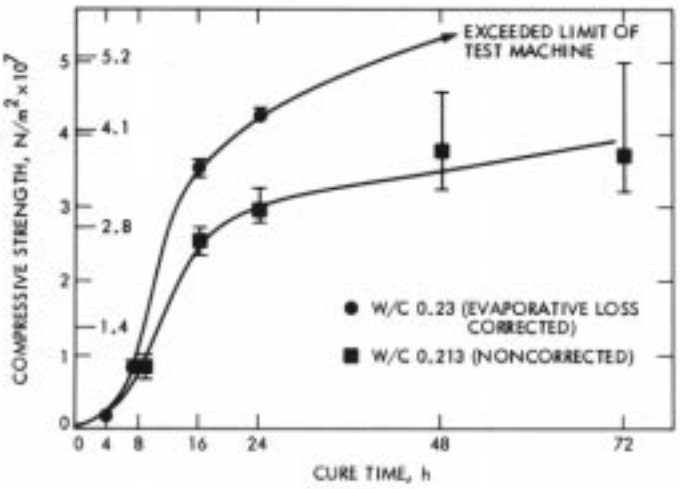
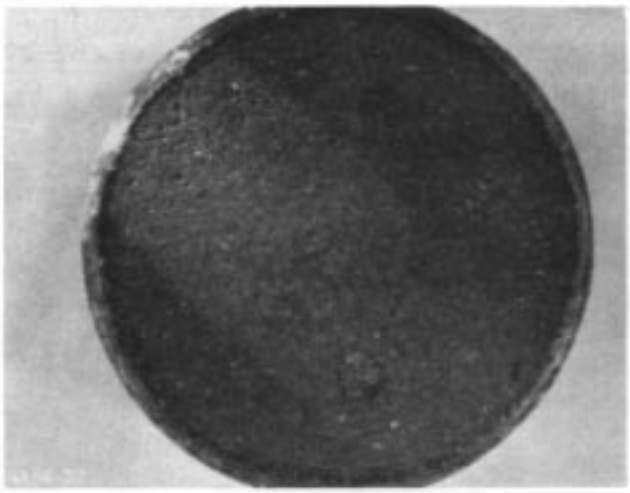
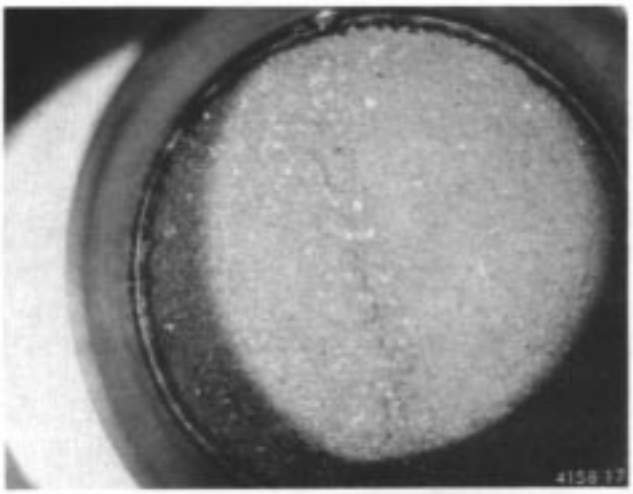



**Fig. 5. Effect of evaporated water loss on the rate of compressive strength development by the dry-pack portland cement grout**



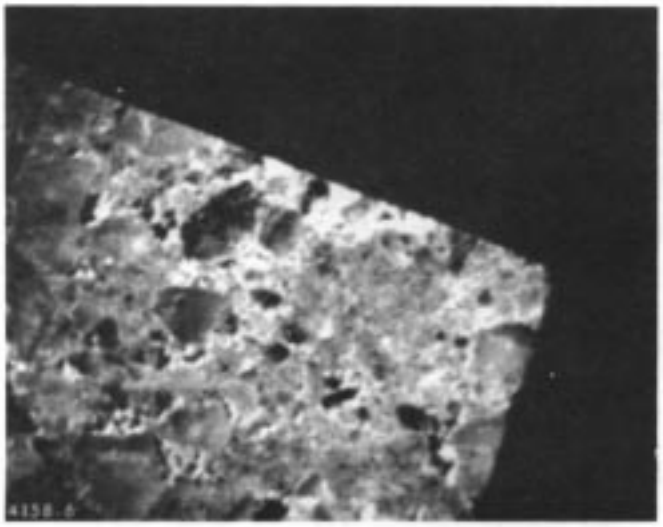
(a) A WATER-CORRECTED DRY-PACK GROUT SURFACE AFTER 15,500 CYCLES SHOWS ONLY MINOR SURFACE PITTING. THE DAMAGED AREAS APPEAR TO BE SUPERFICIAL AND ISOLATED WITH NO EROSION CHANNELS.



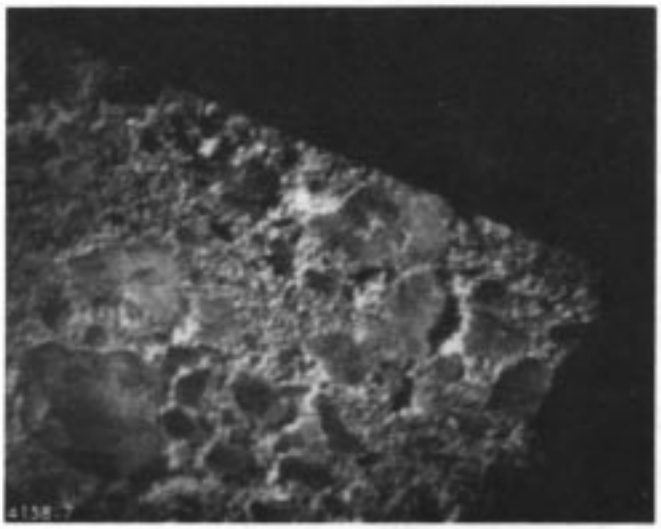
(b) THE DRY-PACK GROUT WITH PACKING INTERFACE, NOW WATER-CORRECTED AND EXPOSED TO OIL AFTER 8 h, SIMULATING PROBABLE REAL CONDITIONS, SHOWS SEVERE EROSION DAMAGE AFTER 5,700 CYCLES.

(c) AFTER 13,700 CYCLES, THE DRY PACKING INTERFACE IS 12.7 mm OR DEEPER AND THE CHANNEL WIDTH HAS UNDERGONE SIMILAR WIDENING. OTHER AREAS OF THE SURFACE HOWEVER, ARE NOT SERIOUSLY DAMAGED.

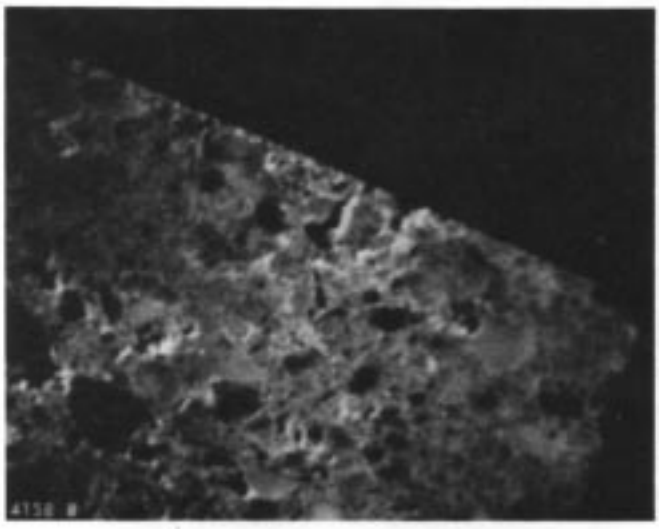
**Fig. 6. While water-corrected grout appears highly durable to erosive effects, its vulnerability to water loss is clearly seen**



(a) CONTROL SPECIMEN BEFORE EXPOSURE.

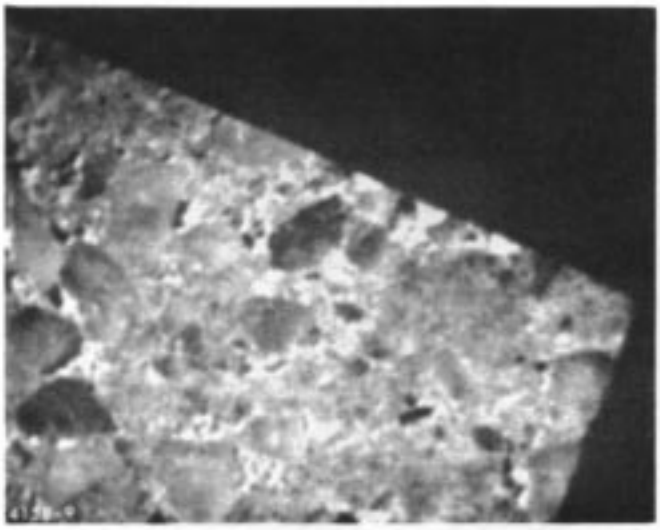


(b) LUBRIZOL EXPOSED SAMPLE SHOWS NO EVIDENCE OF EROSION OR WEIGHT LOSS.

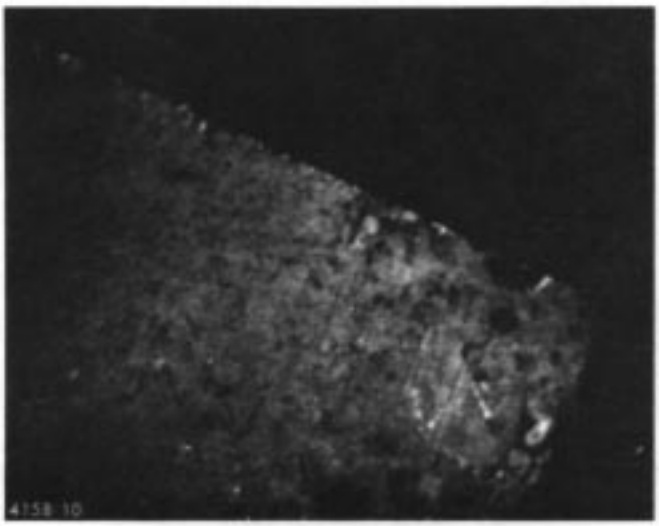


(c) 2, 6-DI-TERT-BUTYL PARACREYSOL SAMPLE, NO EROSION BUT SHOWS WEIGHT LOSS OF 0.85%.

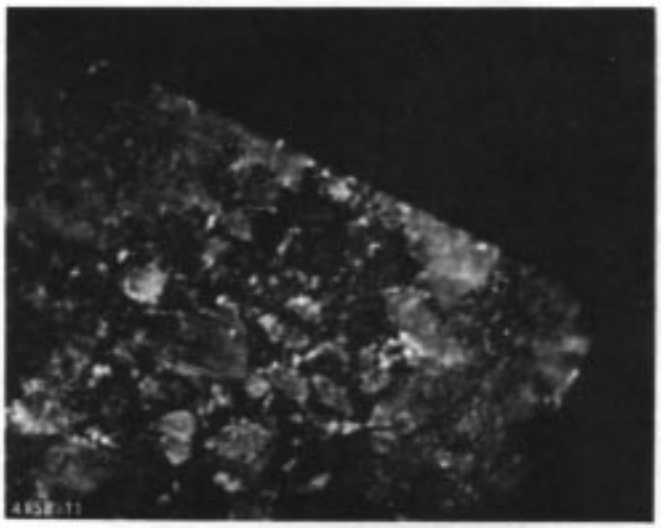
**Fig. 7. Effect of oil additives, I**



(a) CONTROL AFTER EXPOSURE TO THE BEARING OIL. NO WEIGHT LOSS, NO EVIDENCE OF CHEMICAL EROSION.



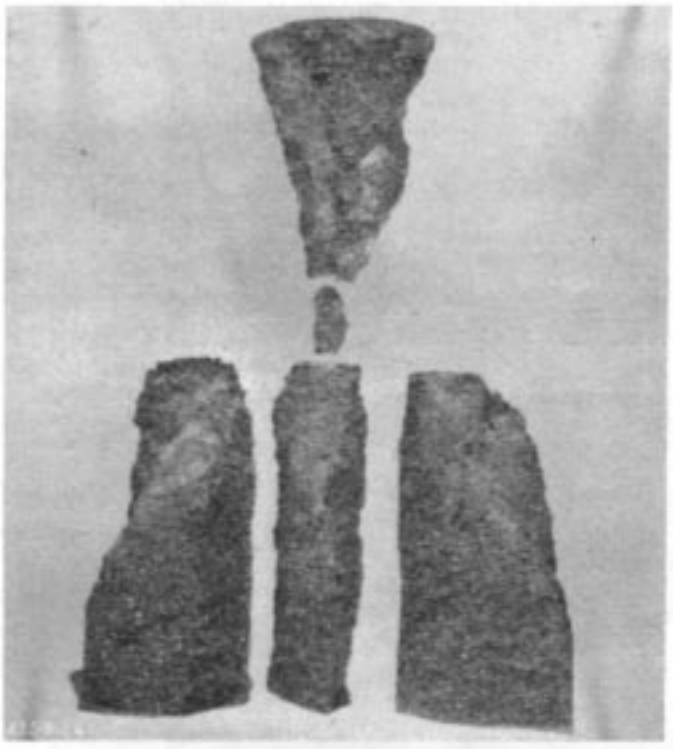
(b) DIPHENYLAMINE - EXPOSURE SAMPLE SHOWS NO WEIGHT LOSS BUT POSSIBLE EVIDENCE OF FRACTURING (CRYSTAL GROWTH) OF THE LARGE AGGREGATES.



(c) PC 1244 POLYMERIC FOAM SUPPRESSANT EXPOSED SAMPLE SHOWS NO EVIDENCE OF DAMAGE.

**Fig. 8. Effect of oil additives, II**

(a) A TYPICAL EPOXY POLYMER CONCRETE CYLINDER IN THE COMPRESSION TEST APPARATUS. THE 7.62-cm × 15.25-cm CYLINDERS WERE GAGED TO MEASURE CHANGES IN THE SPECIMEN LENGTH AND DIAMETER UNDER LOAD.



(b) THE SPECIMENS FAILED CATASTROPHICALLY AND SHOW THE 45° SHEAR FAILURE CHARACTERISTICS TYPICAL OF A HIGH-STRENGTH CONCRETE

**Fig. 9. The epoxy resin test cylinders show high compressive strength and modulus characteristics**

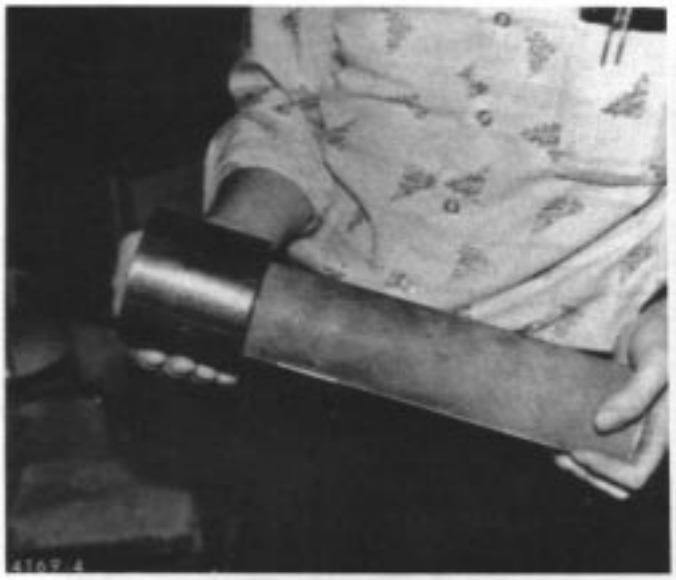


Fig. 10. An erosion test cylinder (dry-pack) with the oil chamber sleeve bonded in place

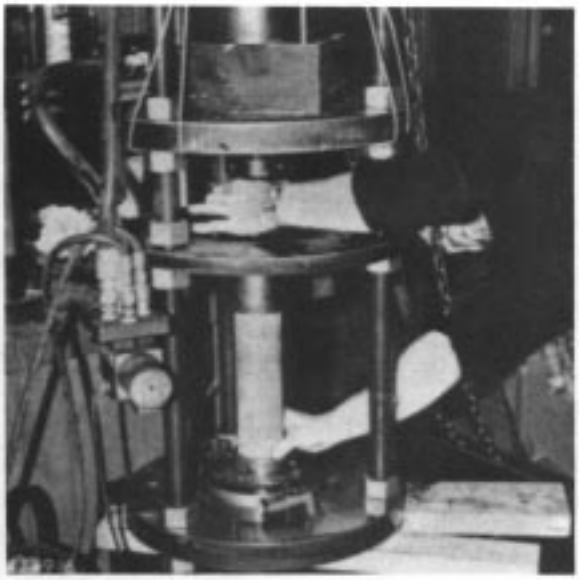


Fig. 12. The piston and sleeve are carefully aligned and the stroke length set so that the piston contacts and exerts full pressure on the cylinder surface in each cycle

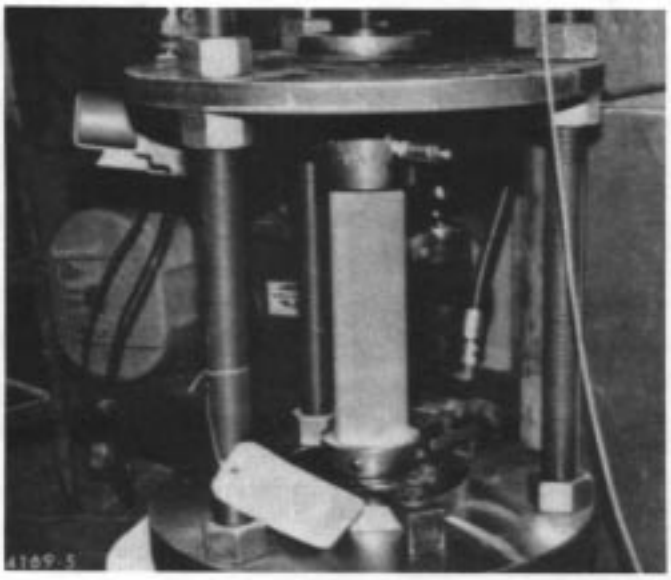
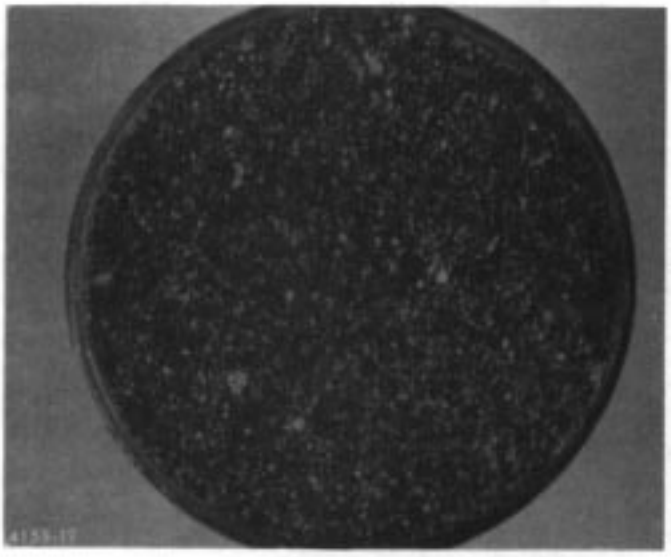
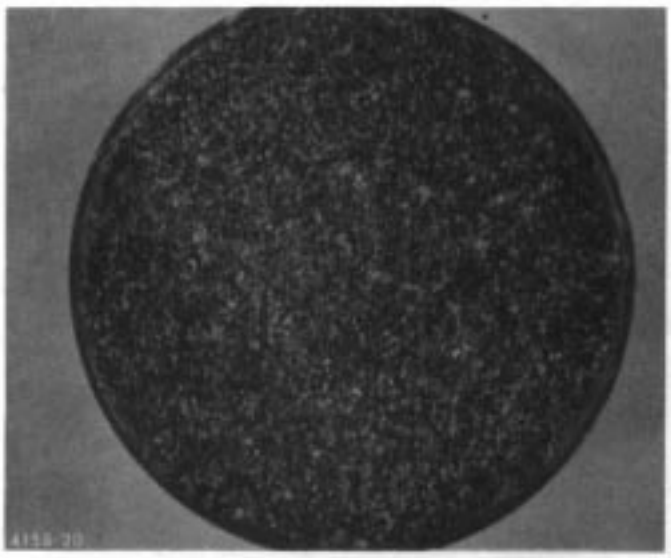


Fig. 11. After inserting the piston into the oil chamber sleeve, the assembly is locked in position by retaining rings top and bottom

Fig. 13. After final checks, the apparatus is lowered into the oil reservoir



(a) EROSION SURFACE OF THE SOLID DESIGN POLYMER CONCRETE SAMPLE AFTER 28,300 CYCLES.



(b) EROSION SURFACE OF THE POROUS POLYMER CONCRETE AFTER 27,500 CYCLES.

**Fig. 14. Polymer concretes showed virtually no sign of erosion or compression damage after more than 27,000 cycles**

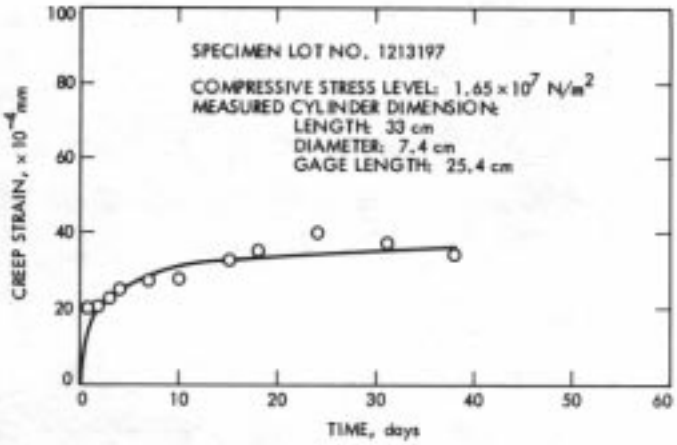



**Fig. 15. Creep of polymer concrete cylinder**

**Fig. 16. Apparatus for testing the curing characteristics of aggregate/resin grouts**

Fig. 17. Epoxy grout containing finely divided silica packed into mold; no evidence of sagging observed

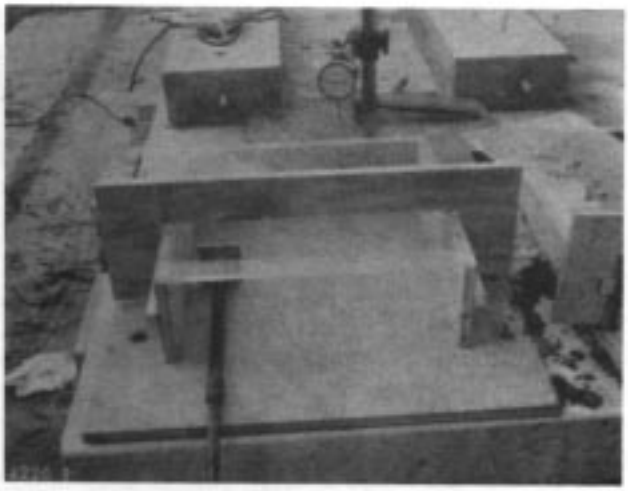


Fig. 19. Test cavity (48 cm × 48 cm × 15 cm) before filling; cavity has wooden sides, bottom, and back, and a plexiglass top; front end is open to permit loading of grout

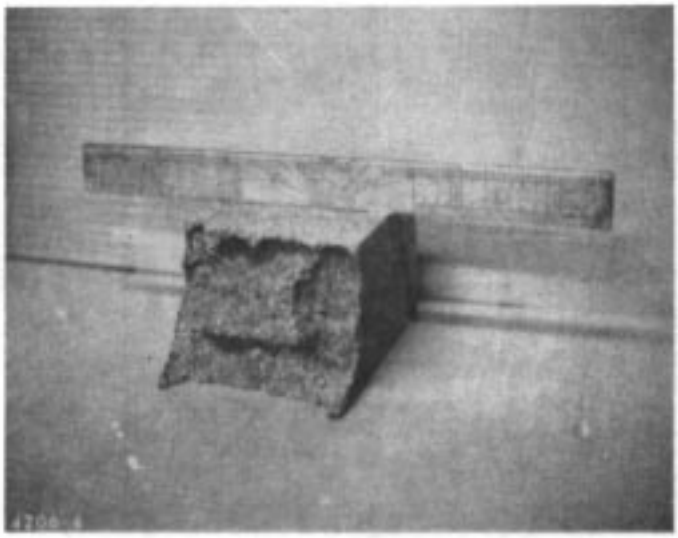
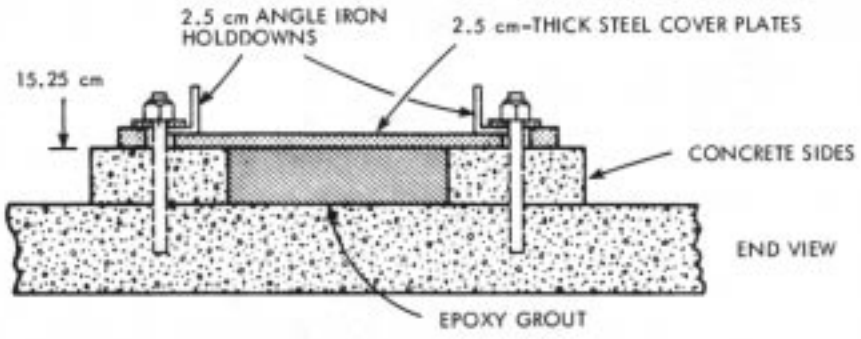


Fig. 18. Epoxy grout after removal from the mold indicating little sag and retention of impressions from tamping

Fig. 20. Loading 48-cm × 48-cm × 15-cm cavity with epoxy grout

**Fig. 21. Partially filled 48-cm × 48-cm × 15-cm cavity; foot of tamper is visible in foreground**



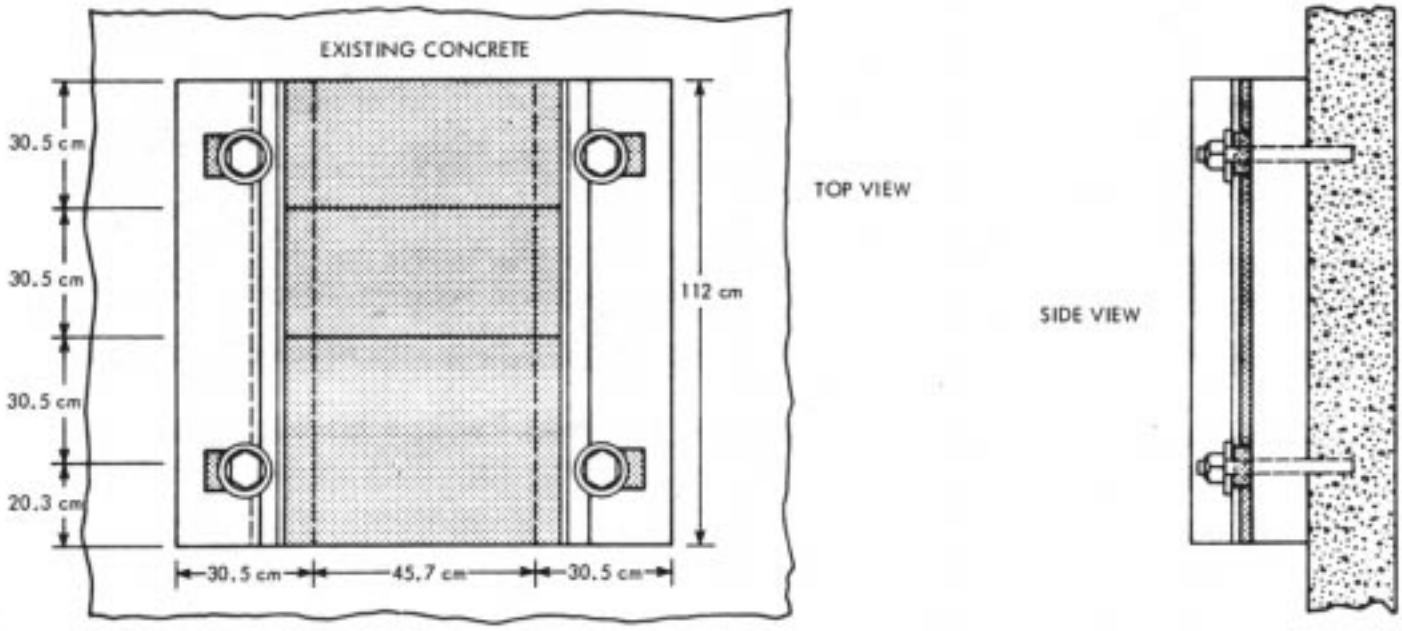
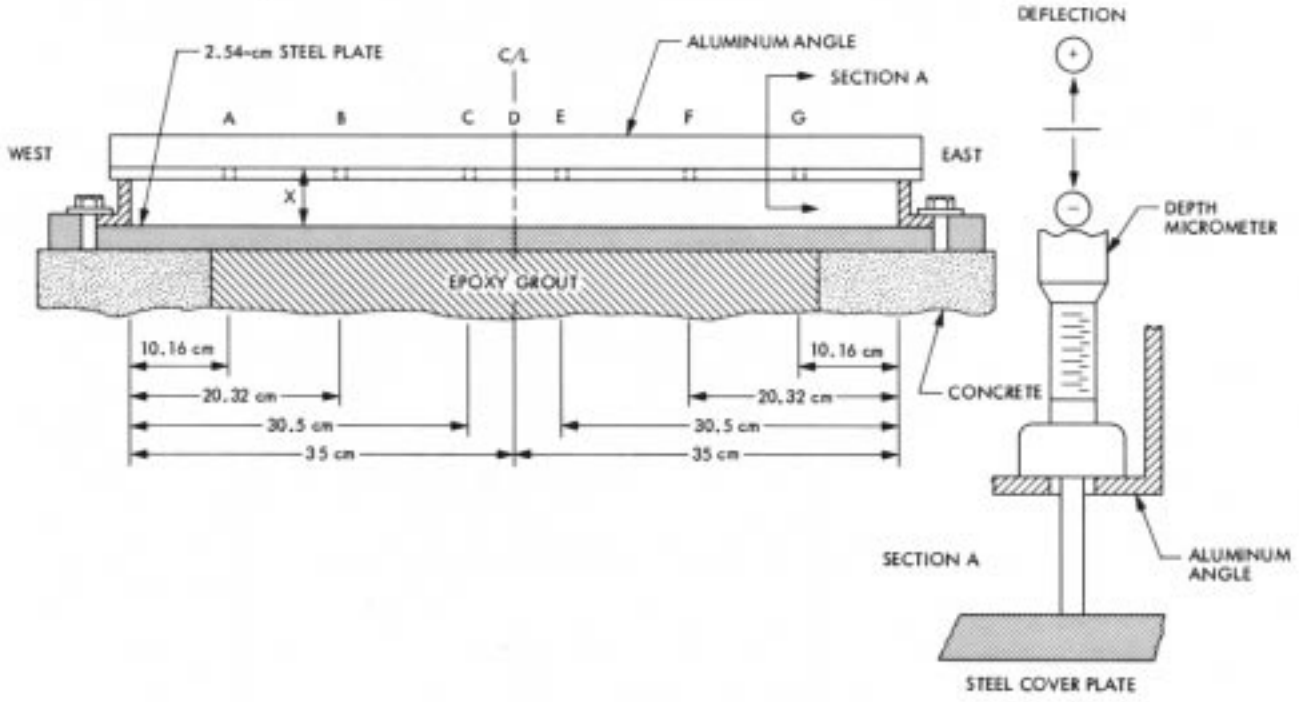




**Fig. 22. Test device for demonstration of epoxy grout**

Fig. 23. Hand mixing of epoxy-TETA resin in 3.785-liter cans




| | | A | B | C | D | E | F | G |
|---|---|---|---|---|---|---|---|---|
| BEFORE TEST | (X) | 2.934 | 2.913 | 2.908 | 2.901 | 2.908 | 2.908 | 2.913 |
| AT MAXIMUM EXOTHERM | (X) | 2.931 | 2.911 | 2.898 | 2.893 | 2.896 | 2.898 | 2.896 |
| Δ | | +0.003 | +0.002 | +0.010 | +0.008 | +0.012 | +0.010 | -0.017 |
| 4-1/2 hours AFTER PLACEMENT | (X) | 2.921 | 2.913 | 2.908 | 2.901 | 2.903 | 2.898 | 2.903 |
| Δ | | +0.013 | +0.000 | +0.000 | +0.000 | +0.005 | +0.010 | +0.010 |
| 24 hours - COLD | (X) | 2.931 | 2.916 | 2.913 | 2.901 | 2.903 | 2.901 | 2.901 |
| Δ | | +0.003 | -0.003 | -0.005 | 0.000 | +0.005 | +0.007 | +0.012 |

DISTANCE X MEASURED WITH DEPTH MICROMETER; DIMENSIONS IN cm

Fig. 24. Apparatus for measuring expansion and contraction of the grout

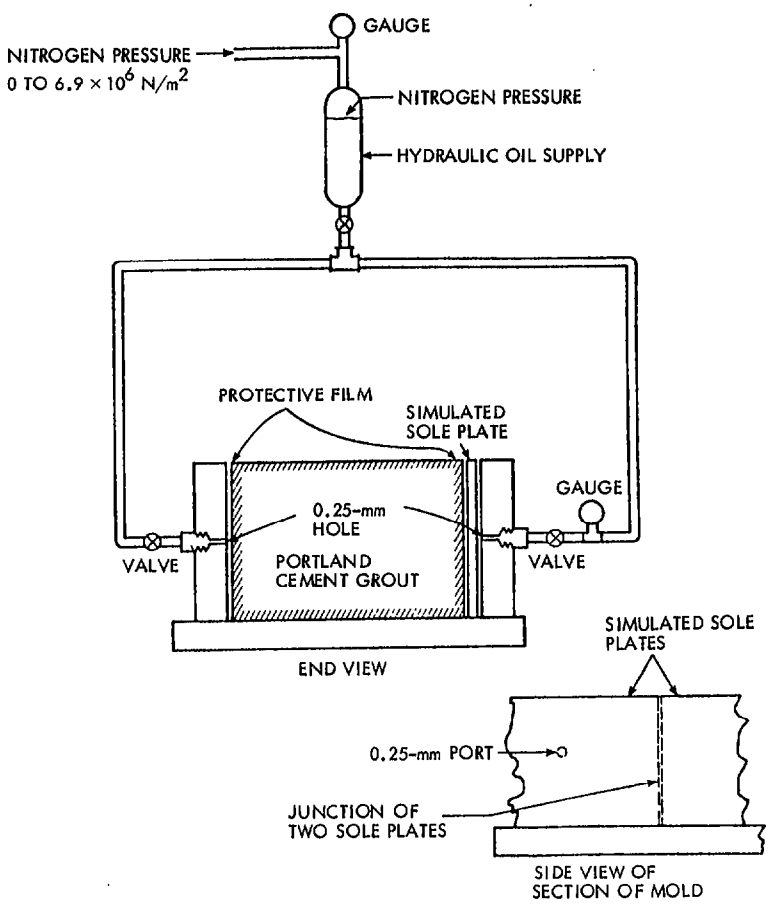



**Fig. 25. Schematic drawing of test rig for evaluation of oil sealing films**